Quality Assurance Project Plan

Supplement B

Base Realignment and Closure Environmental Evaluation Fort Devens, Massachusetts

Submitted to

U.S. Army Environmental
Center (USAEC)
Formerly USATHAMA
Aberdeen Proving Ground, Maryland

Revision 2
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Arthur D. Little, Inc.
Acorn Park
Cambridge, Massachusetts
02140-2390

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Delivery Order 0005

AEC Form 45, 1 Feb 93 replaces THAMA Form 45 which is obsolete.

Table of Contents

List of Figures

List of Tables

List of Acronyms and Abbreviations

See Quality Assurance Project Plan.

1.0 Project Description

1.1 Introduction

See Quality Assurance Project Plan.

1.2 Site Background

1.2.1 Site Description

See Quality Assurance Project Plan.

Tables 1-1B through 1-3B indicate the location of the BRAC EE Study Areas within Fort Devens.

1.2.2 Site History

See Quality Assurance Project Plan.

1.2.3 Previous Investigations

See Quality Assurance Project Plan.

1.3 Task Objectives and Scope of Work

1.3.1 BRAC EE Investigations

The primary objective of this supplemental investigation for the BRAC EE is to evaluate four facility-wide Areas Requiring Environmental Evaluation (AREE) as outlined in the Enhanced Preliminary Assessment, Fort Devens, Massachusetts, April 1992. USATHAMA. The following AREEs will be evaluated:

- Maintenance and Waste Accumulation Areas;
- Transformers;
- Past Spill Site; and
- Previously Removed Underground Storage Tanks.

Tables 1-1B through 1-3B indicate the locations of the study areas within Fort Devens.

The Work Breakdown Structure for the Fort Devens BRAC EE supplemental investigations is presented in Figure 1-1B and the individual Subtasks to complete this task order are described in detail in Supplemental Work Plans submitted February 17, 1994 for each AREE.

Table 1-1B: List of Maintenance and Waste Accumulation Areas

AREE No.	Associated AREE/SA	Building/ Location	Summary of Findings
61A	SA,33,34, 35	Near T-242, 262	Motor pool; no longer in use.
61B	NA	3773/3774	Building 3774 motor pool; in use - rebuilt. Two oil/water separators connected to sanitary sewer. Building 3773 and 3774 have satellite accumulation points for HW.
61C	43F	2021	Motor pool; no longer in use.
61D	NA	1677	Motor pool; in use. Coincides with AREE 61 AP
61E	NA	P-1401	Motor pool; in use - rebuilt. One oil/water separator with discharge to storm drain; HWAA and satellite HW accumulation points.
61F	43C	T-3549	Motor pool; in use.
61G	43G	2008	Motor pool; no longer in use. Present location of gas station.
61H	NA	616 to 618	Building 616 motor pool; in use - rebuilt. Two oil/water separators connected to sanitary sewer; HWAA and satellite accumulation points for HW.
61I	43H and I	601 to 605	Motor pool; in use - rebuilt. One oil/water separator connected to sanitary sewer; HWAA and satellite accumulation points for HW.
61J	NA	612 to 614	Motor pool; in use - rebuilt. One oil/water separator connected to sanitary sewer; HWAA and satellite accumulation for HW.
61K	37	3622	Motor pool; no longer in use.
61L	NA	Across from cemetary	This is a grassy knoll with no historical motor pool. Deleted from study.
61M	37	3606	Motor pool; no longer in use.
61N	NA	T-36705	Motor pool; no longer in use.
61O	43K	2517	Motor pool; in use - rebuilt. One oil/water separator with undetermined outlet. One oil/water separator at carwash facility connected to sanitary sewer.

Table 1-1B: List of Maintenance and Waste Accumulation Areas (continued)

AREE No.	Associated AREE/SA	Building/ Location	Summary of Findings
61P	43L	T-2601	Motor pool; no longer in use.
61Q	43M	2613	Motor pool; no longer in use.
61R	43N, 45	Between 2613 and 2680	Motor pool; no longer in use. One oil/water separator connected to sanitary sewer.
61S	43O	2680	Motor pool; no longer in use.
61T	43P	622	Motor pool; no longer in use.
61U	43Q	Across Street 694	Motor pool; no longer in use.
61V	43S	3412	Motor pool; no longer in use.
61W	49, 37	3601	Motor pool; no longer in use.
61X	38,44,52,57	3713	Three oil/water separators connected to sanitary sewer; HWAA and satellite accumulation points for HW. Only building will be studied.
61Y	NA	3813/3816/3818	Two oil/water separators connected to sanitary sewer; Satellite accumulation points for HW.
61Z	48	202	One oil/water separator connected to sanitary sewer.
61AA	NA	Commissary B-3712	Two oil/water separators in commissary parking lot connected to sanitary sewer.
61AB	NA	219	Satellite accumulation point for HW. One oil/water separator connected to sanitary sewer.
61AC	NA	207	HWAA accumulation point for HW.
61AD	NA	247	Satellite accumulation point for HW.
61AE	NA	1672	HWAA and satellite accumulation points for HW.
61AF	43J	2479/2446	HWAA accumulation point for HW.
61AG	NA	3809	Satellite accumulation point for HW.
61AH	NA	1453	Satellite accumulation point for HW.
61AI	NA	3587	HWAA and satellite accumulation points for HW.
61AJ	NA	3625	Satellite accumulation point for HW.
61AK	NA	P-12	Satellite accumulation point for HW.

Table 1-1B: List of Maintenance and Waste Accumulation Areas (continued)

AREE No.	Associated AREE/SA	Building/ Location	Summary of Findings
61AL	NA	3	Satellite accumulation point for HW.
61AM	NA	3654	Satellite accumulation point for HW.
61AN	NA	2729	Satellite accumulation point for HW.
61AO	2	1450	Satellite accumulation point for HW.
61AP	NA	1677	Deleted from study. Duplicate with 61D.
61AQ	43B	Across from T-3545	Former gas station.
61AR	43D	P-171	Former gas station.
61AS	43E	P-172/2020	Former gas station.
61AT	43R	P-186/696	Former gas station.
61AU	57	3749-3758	Motor pool; in use.
61AV	29	1420, 1417, 1419	Maintenance shops.
61AW	NA	3591	Fire department.
61AX	NA	B-1410	New Commissary. Oil/Water separator; unsure whether connected to sanitary or storm sewers.
61AY	NA	B-1405	POL storage above ground.
61AZ	NA	B-2017	POV wash station. Oil/Water seperators.
61BA	NA	3574	Warehouse
61BB	NA	3411	O'Neil Building.
61BC	NA	3413	USAISD.
61BD	NA	B-216	Contractors' yard at DRMO.

Notes: NA = Not applicable
HW = Hazardous waste
HWAA = Hazardous waste accumulation area for less than 90-day accumulation

Table 1-2B: List of Previously Removed Underground Storage Tanks

Building	Last Product	Gallons	No Contam. Encountered	Contam. Enc. but Removed	Contam. Enc. Not Removed	Contract and FY
0219	Waste Oil	1,000	Unk.			ATEC FY 91/92
0242	Waste Oil	1,000	Unk.			ATEC FY 91/92
0618	#2 Fuel Oil	10,000			X	Jet-Line, FY 91
0618	#2 Fuel Oil	10,000			X	Jet-Line, FY 91
0618	#2 Fuel Oil	5,000			X	Jet-Line, FY 91
0631	#2 Fuel Oil	1,500	Unk.			ATEC FY 91/92
1404	Diesel	5,000	Unk.			ATEC FY 91/92
1419	Kerosene	4,000	Unk.			ATEC FY 91/92
1425	#4 Fuel Oil	10,000	Unk.			ATEC FY 91/92
1429	Mogas	2,500		X		Petr. Tank Svcs., FY 88
1429	Mogas	2,500		X		Petr. Tank Svcs., FY 88
1429	Mogas	1,000		X		Petr. Tank Svcs., FY 88
1429	#2 Fuel Oil	1,000	Unk.			ATEC FY 91/92
1429	Mogas	5,000		X		Petr. Tank Svcs., FY 88
2008	Mogas	10,000			X	Adv. Petr. Prod., FY 91
2008	Mogas	10,000			X	Adv. Petr. Prod., FY 91
2008	Mogas	10,000			X	Adv. Petr. Prod., FY 91
2008	Mogas	10,000			X	Adv. Petr. Prod., FY 91
2419	#2 Fuel Oil	1,000	Unk.			ATEC FY 91/92

Table 1-2B: List of Previously Removed Underground Storage Tanks (continued)

Building	Last Product	Gallons	No Contam. Encountered	Contam. Enc. but Removed	Contam. Enc. Not Removed	Contract and FY
2434	#2 Fuel Oil	1,000	Unk.			ATEC FY 91/92
2452	#2 Fuel Oil	1,000	Unk.			ATEC FY 91/92
2461	#2 Fuel Oil	1,000	Unk.			ATEC FY 91/92
2602	#2 Fuel Oil	1,000			X	Zecco, FY 88
2603	#2 Fuel Oil	1,000	X			Zecco, FY 88
2604	#2 Fuel Oil	1,000	X			Zecco, FY 88
2605	#2 Fuel Oil	1,000		X		Zecco, FY 88
2606	#2 Fuel Oil	1,000		X		Zecco, FY 88
2608	#2 Fuel Oil	1,000	X			Zecco, FY 88
2618	#2 Fuel Oil	1,000		X		Zecco, FY 88
2619	#2 Fuel Oil	1,000			X	Zecco, FY 88
2621	#2 Fuel Oil	1,000	X			Zecco, FY 88
2622	#2 Fuel Oil	1,000	X			Zecco, FY 88
2623	#2 Fuel Oil	1,000	X			Franklin, FY 90
2624	#2 Fuel Oil	1,000		X		Franklin, FY 90
2626	#2 Fuel Oil	1,000		X		Franklin, FY 90
2637	#2 Fuel Oil	1,000		X		Franklin, FY 90
2640	#2 Fuel Oil	1,000	X			Franklin, FY 90
2643	#2 Fuel Oil	1,000		X		Franklin, FY 90
2644	#2 Fuel Oil	1,000		X		Franklin, FY 90
2647	#2 Fuel Oil	1,000		X		Franklin, FY 90
2648	#2 Fuel Oil	1,000		X		Franklin, FY 90
2649	#2 Fuel Oil	1,000		X		Franklin, FY 90
2650	#2 Fuel Oil	1,000			X	Franklin, FY 90
2659	#2 Fuel Oil	1,000		X		Franklin, FY 90

Table 1-2B: List of Previously Removed Underground Storage Tanks (continued)

Building	Last Product	Gallons	No Contam. Encountered	Contam. Enc. but Removed	Contam. Enc. Not Removed	Contract and FY
2660	#2 Fuel Oil	1,000		X		Franklin, FY 90
2661	#2 Fuel Oil	1,000		X		Franklin, FY 90
2662	#2 Fuel Oil	1,000		X		Franklin, FY 90
2686	#2 Fuel Oil	1,000	Unk.			ATEC FY 91/92
2809	#2 Fuel Oil	1,000		X		Franklin, FY 90
3500	#2 Fuel Oil	500	X			Franklin, FY 90
3607	#2 Fuel Oil	5,000		X		Franklin, FY 90
3607	#2 Fuel Oil	5,000		X		Franklin, FY 90
3774	Waste Oil	1,000	Unk.			ATEC FY 91/92
3774	Waste Oil	500	Unk.			ATEC FY 91/92
S-H	#2 Fuel Oil	500			X	Franklin, FY 90
Totals			8	22	11	55

S-H = Shirley Housing

Table 1-3B: Past Spill Sites

Location	Previously Identified SA No.	Date	Chemical	Amount	Report	Status	Remarks
Building 3606-Ramp 3651	NA	3/27/91	Waste Oil	UNK	UNK	Soil has been removed and is awaiting disposal. No known report to MDEP.	
Building 2602	NA	10/30/90	No. 2 Fuel Oil	UNK	Notice	Notice of responsibility sent 11/2/91. No recorded follow-up or cleanup.	
Building 2417	56	6/20/90	No. 2 Fuel Oil	UNK	UNK	Tank removed, some contamination removed.	Deleted from this study, covered in SI
DRMO Yard	32	4/6/90	PCB	UNK	UNK	UNK	Deleted from this study, covered in RI
Bridge 526	59	12/5/90	Lead	UNK	MDEP	Notice of noncompliance issued by MDEP.	Deleted from this study, covered in SI
Building 3412	NA	10/6/89	Diesel Fuel	UNK	UNK	UNK	
South Post	NA	11/19/87	Diesel Fuel	275 gal.	UNK	All contaminated soil removed in drums.	Fuel spill during fueling.
Intel School	NA	UNK	Water Treatment	90 gal.	UNK	UNK	
MAAF-Bldg. 3809	NA	4/9/89	JP-4 Fuel	70 gal.	UNK	Dennis England, OSC, DEH notified. Cleanup complete 4/11/89.	Area was excavated.
Airfield	NA	1/15/89	Helicopter Fuel	15 gal.	UNK	Dennis England notified. Cleanup before any damage occurred.	Fuel spill onto cement floor.
Lake George Street	NA	7/3/90	Diesel	20-30 gal.	MDEP	Notice of responsibility given. No record of follow-up.	Cleanup begun 8/15/90 by Maaci Construction.
Woods	NA	8/15/88	UNK	3 drums	UNK	UNK	
Building 202	48	2/9/89	Oil	UNK	MDEP	Unknown as to whether soil has been treated. No record of follow-up to MDEP.	Deleted from this study, covered in SI
Hospital	NA	1/4/89	Mercury	UNK	UNK	UNK	Mercury from thermometers.
Building 1401	NA	11/14/88	Asphalt	250 gal.	UNK	UNK	
Foxhole near Goddard Memorial	NA	12/7/88	Betz Entec 338	60 gal.	UNK	UNK	
Building 1450	NA	10/27/88	Fuel oil	20 gal.	UNK	UNK	
Pole at corner near Red Cross	NA	9/26/88	PCB oil	<1 lb.	UNK	UNK	
DEH Transformer Storage Area	29	9/14/88	PCB oil	10 gal.	UNK	UNK	Deleted from this study, covered in SI

Table 1-3B: Past Spill Sites (continued)

Location	Previously Identified SA No.	Date	Chemical	Amount	Report	Status	Remarks
Overpass on Barnum Road Near Antietam	NA	1/6/88	Hydraulic oil/gasoline	30 gal.	UNK	UNK	
Elm Street	NA	UNK	JP-5 Fuel	UNK	UNK	JP-5 contaminated fuel.	
Building 2517	NA	10/20/87	Diesel Fuel	20 gal.	UNK	UNK	Motor Asphalt Area.
FD Elementary School	NA	2/26/74	No. 4 Fuel oil	400 gal.	UNK	UNK	

UNK = Unknown
NA = Not Applicable
PCB = Polychlorinated Biphenyls
MDEP = Massachusetts Department of Environmental Protection

1.3.2 BRAC EE Supplemental Investigations

1.3.2.1 BRAC EE Maintenance and Waste Accumulation Areas (AREE 61). The primary objectives of the AREE 61 Maintenance and Waste Accumulation Areas (MWAAs) field investigation are:

- To conduct site investigations at 30 maintenance and waste accumulation areas at Fort Devens to assess the presence of contamination due to unremediated or partially remediated spills or releases of hazardous materials. Three of the 30 MWAAs will be assessed under ongoing Fort Devens Field Investigations. These three sites include 61G, 61I, and 61BG. 61G is included in the SA-43Q Supplemental SI Data Package. 61I is included in the Supplemental SI Data Package (SA43H and 43I) and the Fort Devens UST Management Program 61BG is included in the Fort Devens Installation Solid Waste Disposal Site Closure Assessment.

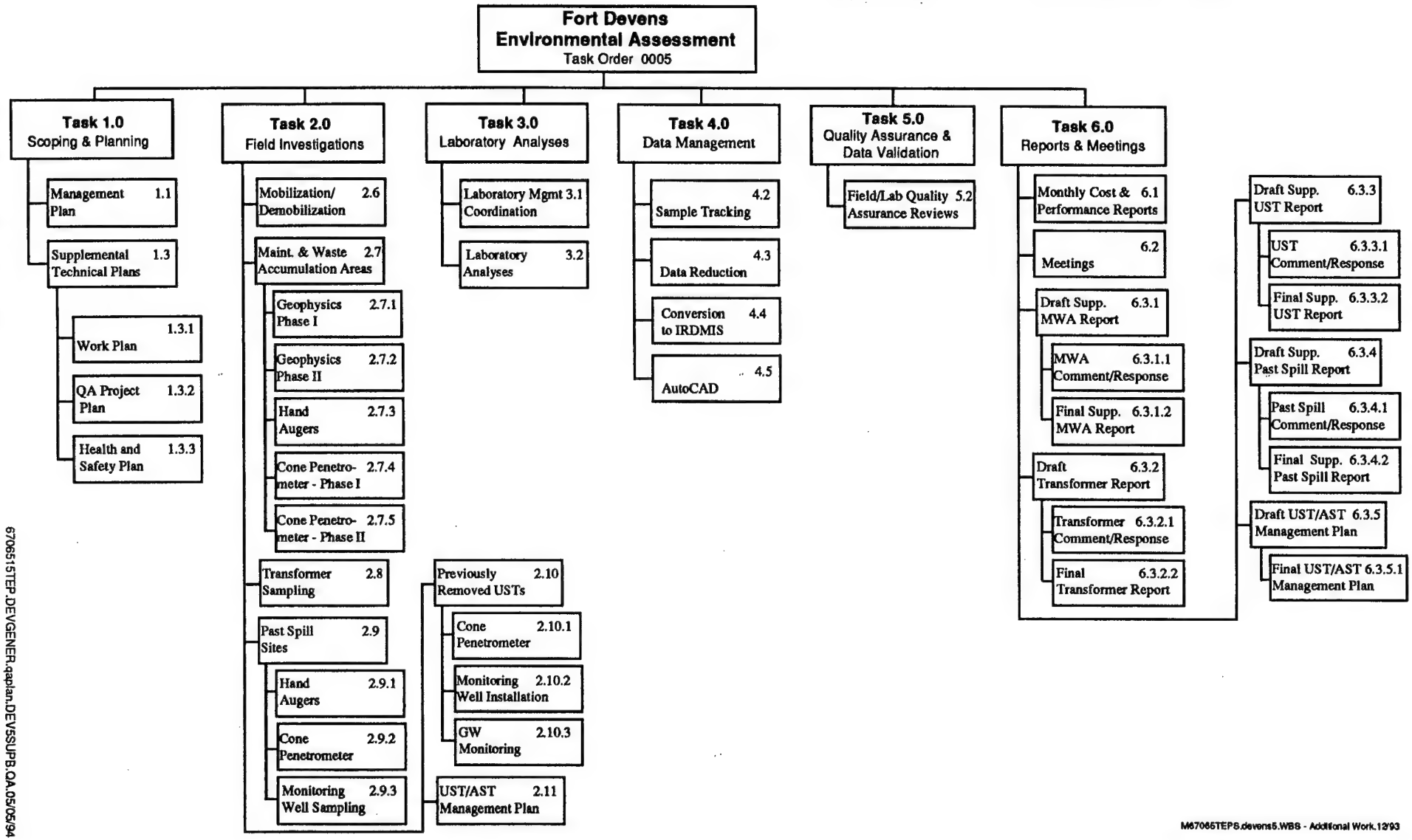
- To evaluate the data to determine which sites require further sampling, or require inclusion in a CERCLA study under the IAG process; and/or are candidates for removal action.

The AREE 61 site investigations will be conducted at 29 areas of the Main Post of Fort Devens. These sites were identified in the *Maintenance and Waste Accumulation Areas Report - AREE 61, BRAC EE, Fort Devens, Massachusetts (USAEC).* This work is being conducted as part of the ongoing Base Realignment and Closure Environmental Evaluation (BRAC EE) at Fort Devens. This investigation has been divided into Phase I and II with 17 sites included in Phase I and 9 sites in Phase II.

1.3.2.2 BRAC EE Previously Removed USTs (AREE 63). The supplemental investigations for AREE 63 addresses sites at Fort Devens which require further action for underground storage tanks that had been removed from the base between 1988 and 1992. Only underground storage tanks removed from the Main and North Posts of the installation were evaluated in this study. A total of 53 previously removed underground storage tank sites were studied under this delivery order. From this study it was determined that 7 require further investigation.

The purpose of this investigation is to evaluate the potential for existing contamination at each of the tank removal locations that require further action. The AREE 63 Memorandum Work Plan dated February 17, 1994 includes discussions of historical data organized by tank removal contractor and then by associated building and tank number. Each site targeted for further investigation is discussed individually and recommendations for further action are provided on a tank-by-tank basis.

Figure 1-1B
Fort Devens BRAC EE Summary Work Breakdown Structure



1.3.2.3 BRAC EE Transformers - Verbeck Gate Substation (AREE 66). The Verbeck Gate substation is located to the northwest of Verbeck Gate on West Main Street Ayer, Massachusetts. This substation is one of the major substations supplying electrical power to Fort Devens. The substation is under the direct control of Fort Devens personnel and is maintained by the installation. Arthur D. Little under contract to the U.S. Army Environmental Center (USAEC contract DAAA15-91-D-0016/0005) investigated historical leaks from equipment containing PCBs. A sampling program was conducted in the summer of 1993 of soils around equipment that either historically or currently contained PCBs which were suspected of leaking to soil. Verbeck Gate Substation was not included in the Sampling Program as part of that study. The current investigation addresses sampling recommended to identify potential PCB spills within the substation.

A total of 41 samples will be taken at Verbeck substation. The samples will be taken in a grid pattern throughout the substation. An increased sampling density will be focused around the transformers and rectifiers where the majority of spills are located. Sample locations will target visual evidence of spills.

1.3.2.4 BRAC EE Past Spill Sites (AREE 69). The primary objectives of the AREE 69 Past Spill Site Study at Fort Devens are:

- To conduct Past Spill Site investigations at 10 areas of Fort Devens to assess the presence of contamination due to unremediated or partially remediated spills of hazardous materials.

- To evaluate the data to determine which sites require further sampling, require inclusion in a CERCLA study under the IAG process; and are candidates for removal action.

The AREE 69 Past Spill Site Study will be conducted at 10 areas of the Main Post and North Post (Moore Army Airfield) BRAC EE of Fort Devens. These sites were identified in the *Draft Past Spill Site Report - AREE 69, BRAC EE, Fort Devens, Massachusetts (USAEC).*

The AREE 69 Work Plan dated February 17, 1994 sets forth the background, plans, and rationale for the activities to be completed during the AREE 69 Past Spill Site Study. The Plan also presents objectives, rationale, frequency, and locations for all sampling occurring in this field investigation.

1.4 Applicability

See Quality Assurance Project Plan.

1.5 Organization of Document

See Quality Assurance Project Plan.

2.0 Project and QA/QC Organization and Responsibilities

See Quality Assurance Project Plan. The organizational structure for the Fort Devens BRAC EE is presented in Figure 2-1B.

2.1 Project Organization

2.1.1 Program Manager
See Quality Assurance Project Plan.

2.1.2 Task Manager
Mr. Richard Waterman is the Arthur D. Little Task Manager for Delivery Order 0005 and will work directly with Dr. Lambe. As Task Manager, his responsibilities include: project staffing and direct management of all staff assigned to Delivery Order 0005; direct financial and schedule control; review and approval of all deliverables; recommending corrective actions, if necessary, to the Program Manager; and maintaining a liaison with the USAEC Project Officer and Fort Devens Environmental Office Manager. In this role, the Task Manager will be responsible for ensuring that the USAEC Project Officer and Fort Devens Environmental Office Manager are kept informed of all technical progress as necessary.

2.1.3 Task Staff
Subtask Managers have been assigned to this project as follows:

- Field Investigation and Deputy Task Manager - Anthony Parkin; and
- Laboratory Analysis and QA/Data Review - Mary Kozik;

2.2 Arthur D. Little QA/QC Organization

See Quality Assurance Project Plan.

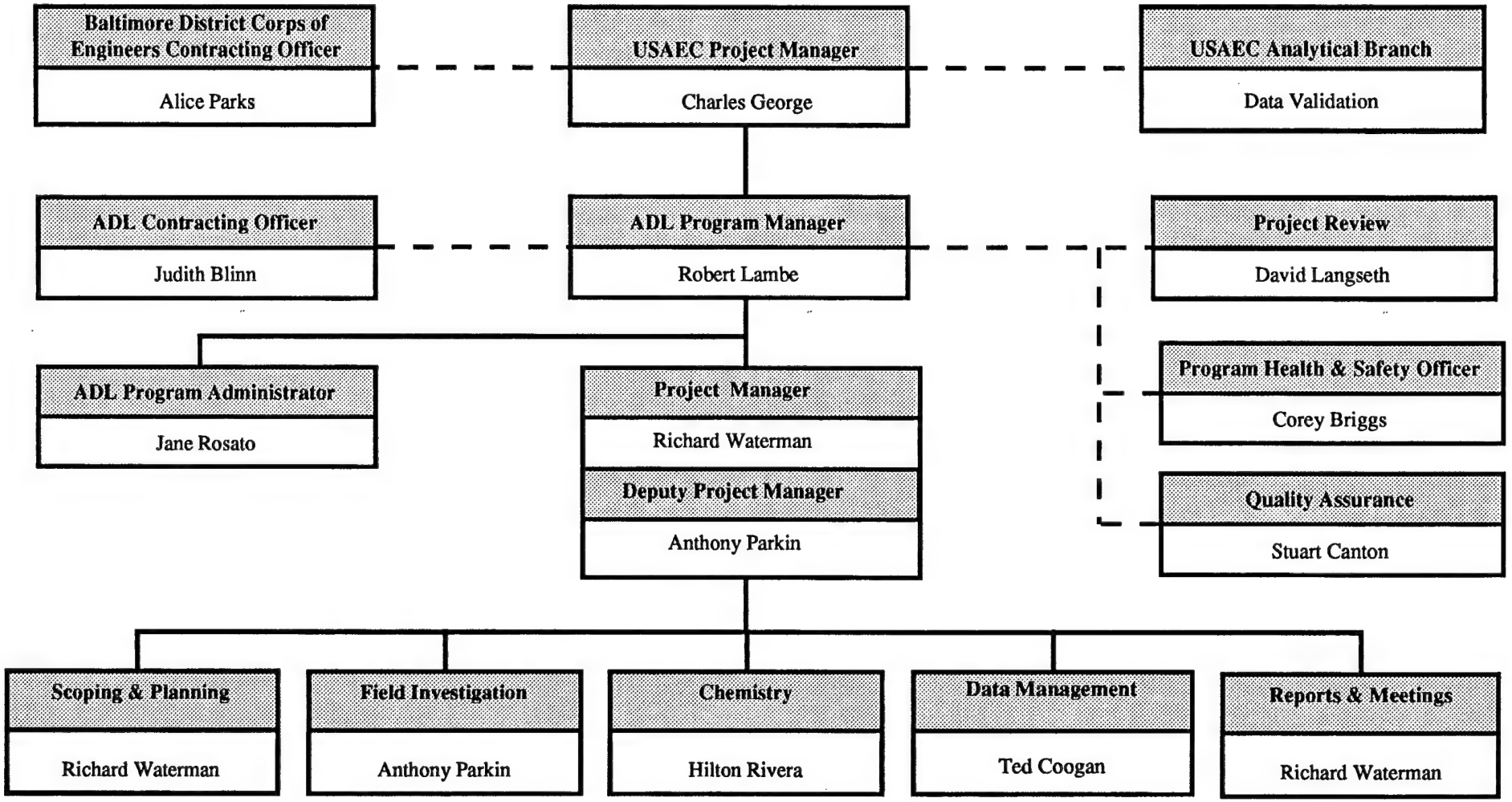
2.2.1 Program QA Officer
See Quality Assurance Project Plan.

2.2.2 Lead Chemist
See Quality Assurance Project Plan.

2.3 DataChem Project QA/QC Organization

See Quality Assurance Project Plan.

Figure 2-1B
Fort Devens Environmental Evaluation - Organizational Chart



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3.0 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Representativeness, Completeness, Comparability

3.1 Introduction

See Quality Assurance Project Plan.

3.2 QA Objectives for Fort Devens Data

See Quality Assurance Project Plan.

USAEC analytical methods are characterized by rigorous QA/QC protocols and documentation requirements. The USAEC data are of high quality, comparable to EPA Level IV data quality (Data Quality Objectives for Remedial Response Activities, USEPA, EPA/540/G-87/003, March 1987). DataChem will use the USAEC approved method for determination of PCBs in surface soil samples. The technique involves extraction of the samples with subsequent analysis of the extract by GC/ECD techniques.

3.2.1 Precision
See Quality Assurance Project Plan.

3.2.2 Accuracy
See Quality Assurance Project Plan.

3.2.3 Representativeness
See Quality Assurance Project Plan.

3.2.4 Completeness
See Quality Assurance Project Plan.

3.2.5 Comparability
See Quality Assurance Project Plan.

4.0 Sample Collection

4.1 Sample Containers, Preservation, and Handling

4.1.1 Sample Containers
See Quality Assurance Project Plan.

4.1.2 Sample Preservation and Holding Times
See Quality Assurance Project Plan.

4.2 Field QC Samples

See Quality Assurance Project Plan.

The type and number of field QC samples to be collected as part of the BRAC EE Investigations are presented in Table 4-1B.

The frequency of field QC samples to be collected as part of the BRAC EE is presented in Table 4-3B. The definitions, rationale, and procedures for collection of field QC samples are provided in the Quality Assurance Project Plan.

4.3 Sample Handling

See Quality Assurance Project Plan.

4.4 Sampling Equipment and Procedures

See Quality Assurance Project Plan.

4.4.1 Test Pit Sampling Procedures
See Quality Assurance Project Plan.

4.4.2 Surface Water Sampling Procedures
See Quality Assurance Project Plan.

4.4.3 Sediment Sampling Procedures
See Quality Assurance Project Plan.

4.4.4 Surface Soil Sampling Procedures
See Quality Assurance Project Plan.

Table 4-1B: BRAC EE Investigations -- Summary of Analytical QC Program

	TCL VOAs	TCL SVOAs	TCL Pest/PCBs	TAL Metals	TPH-IR	TOC	Grain Size	Water Quality Parameters						
								TSS	CL_2/SO_4	TKN	NO_3-NO_2	T-Phosphorus	Hardness	Alk
Total Aqueous Samples	70	70	0	10	70	0	0	60	60	60	60	60	60	60
Field Dup.	4	4	0	1	4	0	0	3	3	3	3	3	3	3
Matrix Spike	4	4	0	1	0	0	0	0	3	3	3	3	3	3
Matrix Spike Dup.	4	4	0	1	0	0	0	0	3	3	3	3	3	3
Field Blank	8	8	0	8	8	0	0	8	8	8	8	8	8	8
Rinsate Blank[1]	4	4	0	4	4	0	0	4	4	4	4	4	4	4
Trip Blank[2]	16	0	0	0	0	0	0	0	0	0	0	0	0	0
Total QC Samples	40	24	0	15	16	0	0	15	21	21	21	21	21	21
Total Solids Samples	134	134	11	90	123	70	134	0	0	0	0	0	0	0
Field Dup.	7	7	1	5	6	4	0	0	0	0	0	0	0	0
Matrix Spike	7	7	1	5	0	4	0	0	0	0	0	0	0	0
Matrix Spike Dup.	7	7	1	5	0	4	0	0	0	0	0	0	0	0
Field Blank	8	8	8	8	8	8	0	0	0	0	0	0	0	0
Rinsate Blank[1]	4	4	4	4	4	4	0	0	0	0	0	0	0	0
Trip Blank[2]	16	0	0	0	0	0	0	0	0	0	0	0	0	0
Total QC Samples	49	33	15	27	18	24	0	0	0	0	0	0	0	0

1. These numbers were based on the assumption an average of 2 field crews at the site every week.

2. There should be a trip blank on every shipment containing VOA samples. These numbers were based on the assumption of two shipments per week.

Table 4-2B: Frequency of Field Quality Control Samples

Field Blank:	One per event[1], these will only be collected when sampling aqueous matrices; ASTM Type I deionized water or equivalent used for organic field blanks; distilled, deionized water used for inorganic field blanks.
Equipment/ Rinsate Blank:	One at a 10% frequency[2] for all parameters except TOC and Grain Size; ASTM Type I deionized water or equivalent used for organic rinsate blanks; distilled, deionized water used for inorganic rinsate blanks.
Trip Blank:	For volatile organic analyses; minimum 10% frequency[2], Purged deionized ASTM Type I deionized water or equivalent is to be used for trip blanks.
Field Duplicate:	One per 20 samples or 5% per matrix for all parameters except TOC and Grain Size.
Matrix Spike/ Matrix Spike Duplicate:	Metals and Pesticides/PCBs analysis only: one per every 20 samples or 5% per matrix.

1 = Assumes one field blank is required for the entire sample event.
2 = Approximate,may vary depending upon sampling events, to be used for estimating purposes.

4.4.5 Concrete/Asphalt Chip Sampling Procedures
See Quality Assurance Project Plan.

4.4.6 Soil Boring Procedures
See Quality Assurance Project Plan.

4.4.6.1 Subsurface Clearance Program. See Quality Assurance Project Plan.

4.4.7 Ground Water Sampling Procedures
See Quality Assurance Project Plan.

4.4.8 Wipe Sampling Procedures
See Quality Assurance Project Plan.

4.4.9 Sample Location and Elevation Survey Procedures
See Quality Assurance Project Plan.

4.4.10 Investigation-Derived Waste Handling Procedures
See Quality Assurance Project Plan.

4.4.11 Geoprobe® Sampling
See Quality Assurance Project Plan.

5.0 Sample Custody

5.1 Field Custody Procedures

See Quality Assurance Project Plan.

Figure 5-1B summarizes the site identification and field numbering system to be applied to all samples collected during completion of the BRAC EE Investigation.

5.2 Laboratory Custody Procedures

See Quality Assurance Project Plan.

Figure 5-1B: Site Identification and Field Sample Numbering System
Fort Devens BRAC EE Investigation

SITE IDENTIFICATION CODE (10 Characters)									
CHARACTER NUMBER									
1	2	3	4	5	6	7	8	9	10
AREE Number: 61XX 63XX 66XX 69XX				Hyphen	Year of Activity		Hyphen	Site Number: 1-99	

QC SAMPLE IDENTIFICATION CODE (8 CHARACTERS)							
CHARACTER NUMBER							
1	2	3	4	5	6	7	8
Media Type: B - Soil Drilling M - Ground Water Well S - Soil/Hand Auger F - Soil/Geoprobe® G - Ground Water/Geoprobe®	Sample Type: X - Regular T - Trip Blank F - Field Blank R - Rinse Blank D - Duplicate Sample M - Matrix Spike Z - Matrix Spike Duplicate	Event Code: C - Spring 1994 D - Summer 1994	AREE Number: 1, 3, 6, 9	AREE Code: (Characters 3-4 from Site ID)		Sample Replicate Number: 0-9 A-Z	Sample Depth: 0 - 0.0 feet 2 - 2.0 feet 4 - 4.0 feet S - Shallow D - Deep L - Lower M - Middle U - Upper W - Sample Collected at Water Table X - Water/No Depth

6.0 Calibration Procedures and Frequency

6.1 Field Instrumentation

See Quality Assurance Project Plan.

6.2 Laboratory Calibration

See Quality Assurance Project Plan.

6.2.1 Laboratory Instrumentation Calibration

6.2.1.1 Calibration Standards. See Quality Assurance Project Plan.

6.2.1.2 Calibration Frequency. See Quality Assurance Project Plan.

6.2.1.3 Tuning and GC/MS Mass Calibration. See Quality Assurance Project Plan.

6.2.1.4 Decafluorotriphenylphosphine (DFTPP). See Quality Assurance Project Plan.

6.2.1.5 p-Bromofluorobenzene (BFB). See Quality Assurance Project Plan.

6.2.2 Operational Calibration

See Quality Assurance Project Plan.

6.2.2.1 General Calibration Procedures. See Quality Assurance Project Plan.

6.2.2.2 Method Blank. See Quality Assurance Project Plan.

6.2.2.3 Calibration Curve. See Quality Assurance Project Plan.

6.2.3 Calibration for USAEC Approved Methods

See Quality Assurance Project Plan.

7.0 Analytical Procedures

7.1 Analytical Program

See Quality Assurance Project Plan.

7.2 Laboratory Method Approval

See Quality Assurance Project Plan.

7.2.1 Laboratory Methods Requiring Approval

See Quality Assurance Project Plan.

7.2.2 Methods Not Requiring Approval

See Quality Assurance Project Plan.

7.3 Analyst Qualification

See Quality Assurance Project Plan.

7.4 Analytical Methods

See Quality Assurance Project Plan.

7.4.1 Sulfate and Chloride

See Quality Assurance Project Plan.

7.4.2 Volatile Organics (GC/MS)

See Quality Assurance Project Plan.

7.4.3 Semivolatile (Acid/Base/Neutral) Organics (GC/MS)

See Quality Assurance Project Plan.

7.4.4 Organochlorine Pesticides/PCBs (GC/ECD)

See Quality Assurance Project Plan.

7.4.5 Metals

7.4.5.1 Inductively Coupled Plasma Atomic Emission Spectroscopy (ICAP). See Quality Assurance Project Plan.

7.4.5.2 Cold Vapor (Mercury). See Quality Assurance Project Plan.

7.4.5.3 Graphite Furnace Atomic Absorption. See Quality Assurance Project Plan.

7.4.6 Explosives
See Quality Assurance Project Plan.

7.4.7 TSS (Total Suspended Solids)
See Quality Assurance Project Plan.

7.4.8 TPHC (Total Petroleum Hydrocarbons by Infrared)
See Quality Assurance Project Plan.

7.4.9 TOC in Sediment by IR
See Quality Assurance Project Plan.

7.4.10 Total Phosphorous and Phosphate
See Quality Assurance Project Plan.

7.4.11 Total Kjeldahl Nitrogen (TKN) in Water by Automated Spectrophotometry
See Quality Assurance Project Plan.

7.4.12 Organophosphorus Pesticides
See Quality Assurance Project Plan.

7.4.13 Chlorinated Herbicides
See Quality Assurance Project Plan.

7.4.14 Nitrate
See Quality Assurance Project Plan.

7.4.15 Hardness
See Quality Assurance Project Plan.

7.4.16 Alkalinity
See Quality Assurance Project Plan.

7.4.17 Asbestos (Bulk) by Polarizing Light Microscopy
See Quality Assurance Project Plan.

7.4.18 Particle Size by Sieve Analysis
See Quality Assurance Project Plan.

7.4.19 TCLP Leachate Preparation
See Quality Assurance Project Plan.

7.4.20 Volatile Organic Compounds (VOCs) in Ambient Air
See Quality Assurance Project Plan.

7.4.21 Metals in Ambient Air
See Quality Assurance Project Plan.

7.4.22 PCB in Ambient Air
See Quality Assurance Project Plan.

7.4.23 Sediment and Surface Water Bioassays
See Quality Assurance Project Plan.

7.5 Field Analytical Methods

See Quality Assurance Project Plan.

7.5.1 Total Petroleum Hydrocarbons by Non-Dispersive Infrared Spectrometry (NDIR)
See Quality Assurance Project Plan.

7.5.2 BTEX (Benzene, Toluene, Ethylbenzene and Xylene)
See Quality Assurance Project Plan.

7.5.3 PCBs Using Immunosorbent Assay (Immunoassay)
See Quality Assurance Project Plan.

7.5.4 TNT Explosives Using Immunosorbent Assay (Immunoassay)
See Quality Assurance Project Plan.

8.0 Data Reduction, Validation, and Reporting

8.1 Arthur D. Little's Data Management

See Quality Assurance Project Plan.

8.1.1 Flow of Map Data into the IRDMIS

See Quality Assurance Project Plan.

8.1.2 Flow of Geotechnical Data into the IRDMIS

See Quality Assurance Project Plan.

8.1.3 Flow of Chemical Data into the IRDMIS

See Quality Assurance Project Plan.

8.2 Data Reduction

See Quality Assurance Project Plan.

8.3 Data Validation

See Quality Assurance Project Plan.

8.3.1 USAEC Data Validation Procedures

See Quality Assurance Project Plan.

8.3.2 USEPA Data Validation Procedures

See Quality Assurance Project Plan.

8.4 IRDMIS Record and Group Checks

See Quality Assurance Project Plan.

8.5 Data Reporting

See Quality Assurance Project Plan.

9.0 Internal QC Checks and Frequency

9.1 Control Samples

See Quality Assurance Project Plan.

9.2 Field Control Samples

See Quality Assurance Project Plan.

9.2.1 Trip Blanks
See Quality Assurance Project Plan.

9.2.2 Field Equipment/Rinsate Blanks
See Quality Assurance Project Plan.

9.2.3 Field Duplicates
See Quality Assurance Project Plan.

9.2.4 Field Blanks
See Quality Assurance Project Plan.

9.3 Laboratory Control Samples

See Quality Assurance Project Plan.

9.3.1 Laboratory Blanks
See Quality Assurance Project Plan.

9.3.2 Laboratory Duplicates
See Quality Assurance Project Plan.

9.3.3 Calibration Standards
See Quality Assurance Project Plan.

9.3.4 Spike Sample
See Quality Assurance Project Plan.

9.3.5 Internal Standard
See Quality Assurance Project Plan.

9.4 Concentration and Frequency of Control Samples

See Quality Assurance Project Plan.

9.4.1 Class 1 Certified Method

See Quality Assurance Project Plan.

9.4.2 Class 1A Certified Method (GC/MS only)

See Quality Assurance Project Plan.

9.5 Data Reporting for QC

9.5.1 Class 1, Class 1A, and Class 1B Certified Methods

See Quality Assurance Project Plan.

10.0 Performance and System Audits

10.1 Field Audits

See Quality Assurance Project Plan.

10.2 Laboratory Audits

See Quality Assurance Project Plan.

10.2.1 Data Review

See Quality Assurance Project Plan.

10.3 Project Audits

See Quality Assurance Project Plan.

11.0 Preventive Maintenance

11.1 Field Instruments

See Quality Assurance Project Plan.

11.2 Laboratory Equipment

See Quality Assurance Project Plan.

12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

12.1 Lack of Fit (LOF) and Zero Intercept (ZI) Tests

See Quality Assurance Project Plan.

12.2 Certified Reporting Limit (CRL)

See Quality Assurance Project Plan.

12.3 Method Certification Accuracy

See Quality Assurance Project Plan.

12.4 Method Certification Standard Deviation

See Quality Assurance Project Plan.

12.5 Method Certification Percent Inaccuracy

See Quality Assurance Project Plan.

12.6 Method Certification Percent Imprecision

See Quality Assurance Project Plan.

12.7 Data Moving-Average Accuracy and Precision

See Quality Assurance Project Plan.

12.8 Control Charts

See Quality Assurance Project Plan.

12.8.1 Control Chart Plotting: Single-Day

See Quality Assurance Project Plan.

12.8.2 Three-Point Moving Average

See Quality Assurance Project Plan.

12.9 Out-of-Control Conditions

See Quality Assurance Project Plan.

12.10 Non-USAEC Methods

See Quality Assurance Project Plan.

12.11 Completeness

See Quality Assurance Project Plan.

13.0 Corrective Actions

13.1 Field Situations

See Quality Assurance Project Plan.

13.2 Laboratory Situations

See Quality Assurance Project Plan.

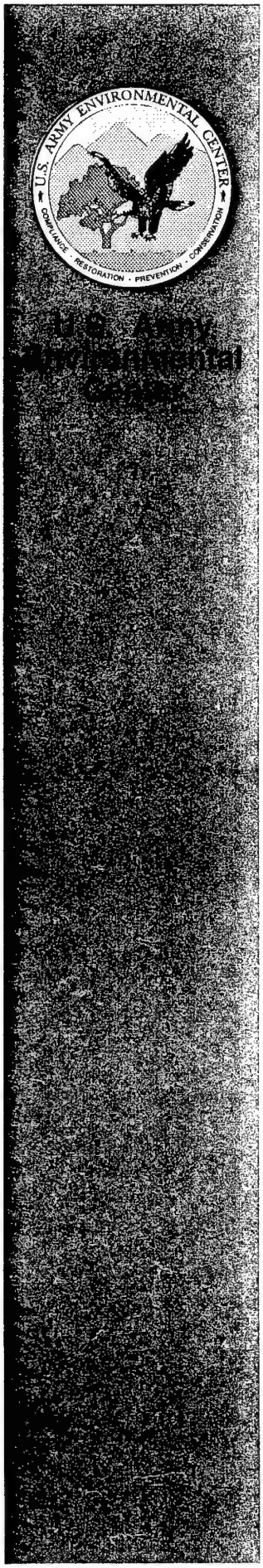
14.0 Quality Assurance Reports to Management

14.1 Laboratory Reports

See Quality Assurance Project Plan.

14.2 Program QA Officer and Lead Chemist Reports

See Quality Assurance Project Plan.



Quality Assurance Project Plan

Supplement C

Base Realignment and Closure Environmental Evaluation Fort Devens, Massachusetts

Submitted to

U.S. Army Environmental Center (USAEC)
Formerly USATHAMA
Aberdeen Proving Ground, Maryland

Revision 1
November 14, 1994

Arthur D. Little, Inc.
Acorn Park
Cambridge, Massachusetts
02140-2390

ADL Reference 67071-14

DAAA15-91-D-0016
Delivery Order 0011

AEC Form 45, 1 Feb 93 replaces THAMA Form 45 which is obsolete.

Quality Assurance Project Plan

Supplement C

Base Realignment and Closure Environmental Evaluation Fort Devens, Massachusetts

Program Manager, Robert Lambe — Date: 11-16-94

Task Manager, Richard Waterman — Date: 11/14/94

Program Quality Assurance Officer, Stuart Canton — Date

Submitted to

U.S. Army Environmental Center (USAEC)
Formerly USATHAMA
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Arthur D Little

Table of Contents

List of Appendices

List of Figures

List of Acronyms and Abbreviations

See Quality Assurance Project Plan.

N/A	Not Applicable

1.0 Project Description

1.1 Introduction

See Quality Assurance Project Plan.

1.2 Site Background

1.2.1 Site Description

See Quality Assurance Project Plan.

Table 2-1 of the Draft Supplemental Work Plan for Fort Devens BRAC EE, Part II indicates the location of the BRAC EE Study Areas within Fort Devens.

1.2.2 Site History

See Quality Assurance Project Plan.

1.2.3 Previous Investigations

See Quality Assurance Project Plan.

1.3 Task Objectives and Scope of Work

The primary objective of this BRAC EE is to evaluate those areas on the Main and North Post that have the potential for containing asbestos, lead based paint and/or radon.

This evaluation will involve a review of documentation for each site, interviews with key Fort Devens personnel, and site tours. Asbestos and lead based paint samples will be collected.

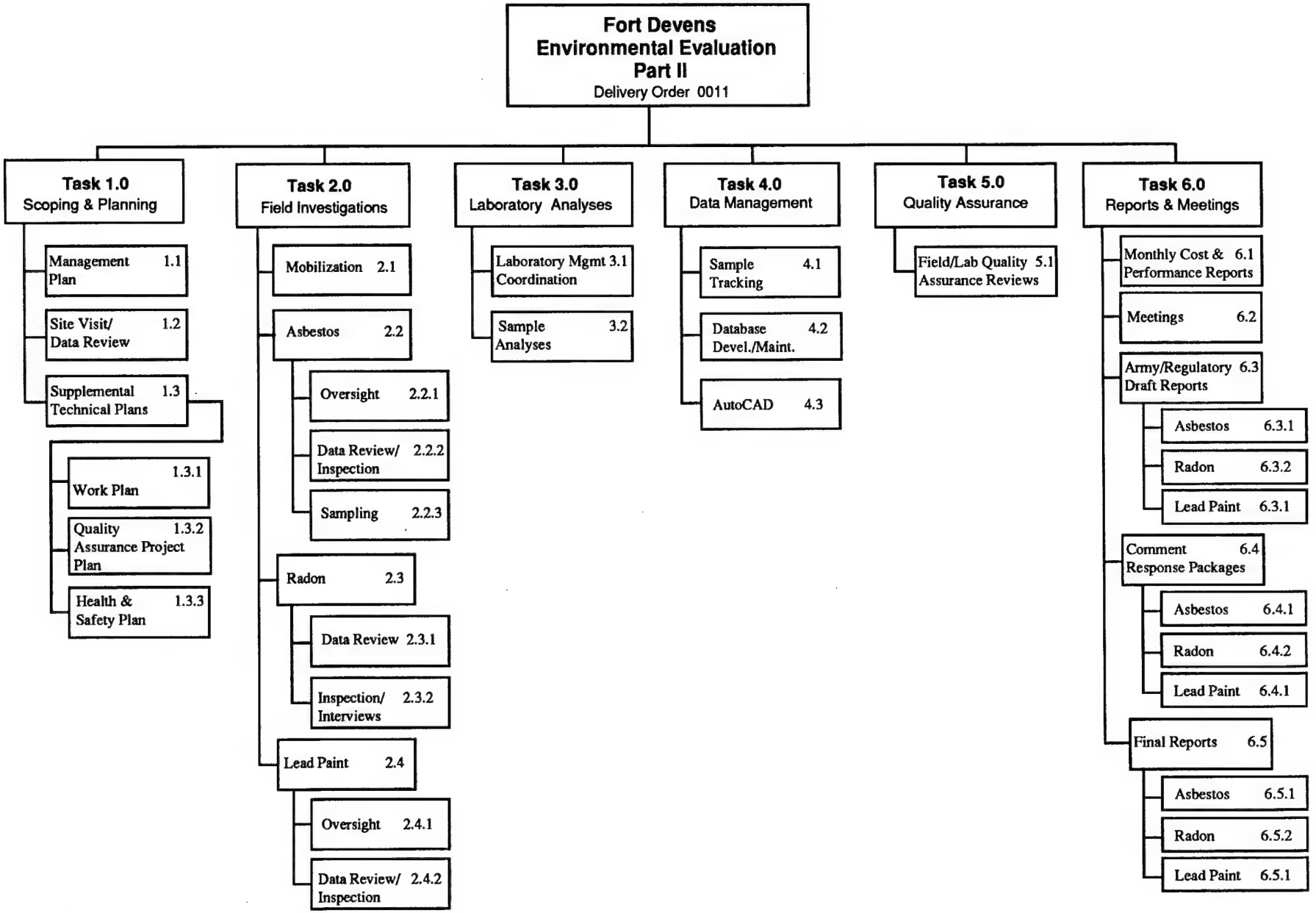
1.4 Applicability

See Quality Assurance Project Plan.

1.5 Organization of Document

See Quality Assurance Project Plan.

Figure 1-1C: Fort Devens BRAC EE Part II Summary Work Breakdown Structure



2.0 Project and QA/QC Organization and Responsibilities

See Quality Assurance Project Plan. The organizational structure for the Fort Devens BRAC EE is presented in Figure 2-1C.

2.1 Project Organization

2.1.1 Program Manager
See Quality Assurance Project Plan.

2.1.2 Task Manager
Mr. Richard Waterman is the Arthur D. Little Task Manager for Delivery Order 0011 and will work directly with Dr. Lambe. As Task Manager, his responsibilities include: project staffing and direct management of all staff assigned to Delivery Order 0011; direct financial and schedule control; review and approval of all deliverables; recommending corrective actions, if necessary, to the Program Manager; and maintaining a liaison with the USAEC Project Officer and Fort Devens Environmental Office Manager. In this role, the Task Manager will be responsible for ensuring that the USAEC Project Officer and Fort Devens Environmental Office Manager are kept informed of all technical progress as necessary.

2.1.3 Task Staff
Subtask Managers have been assigned to this project as follows:

- Field Investigation and Deputy Task Manager - Erica Cahill
- Laboratory Analysis and QA/Data Review - Hilton Rivera

2.2 Arthur D. Little QA/QC Organization

See Quality Assurance Project Plan.

2.2.1 Program QA Officer
See Quality Assurance Project Plan.

2.2.2 Lead Chemist
See Quality Assurance Project Plan.

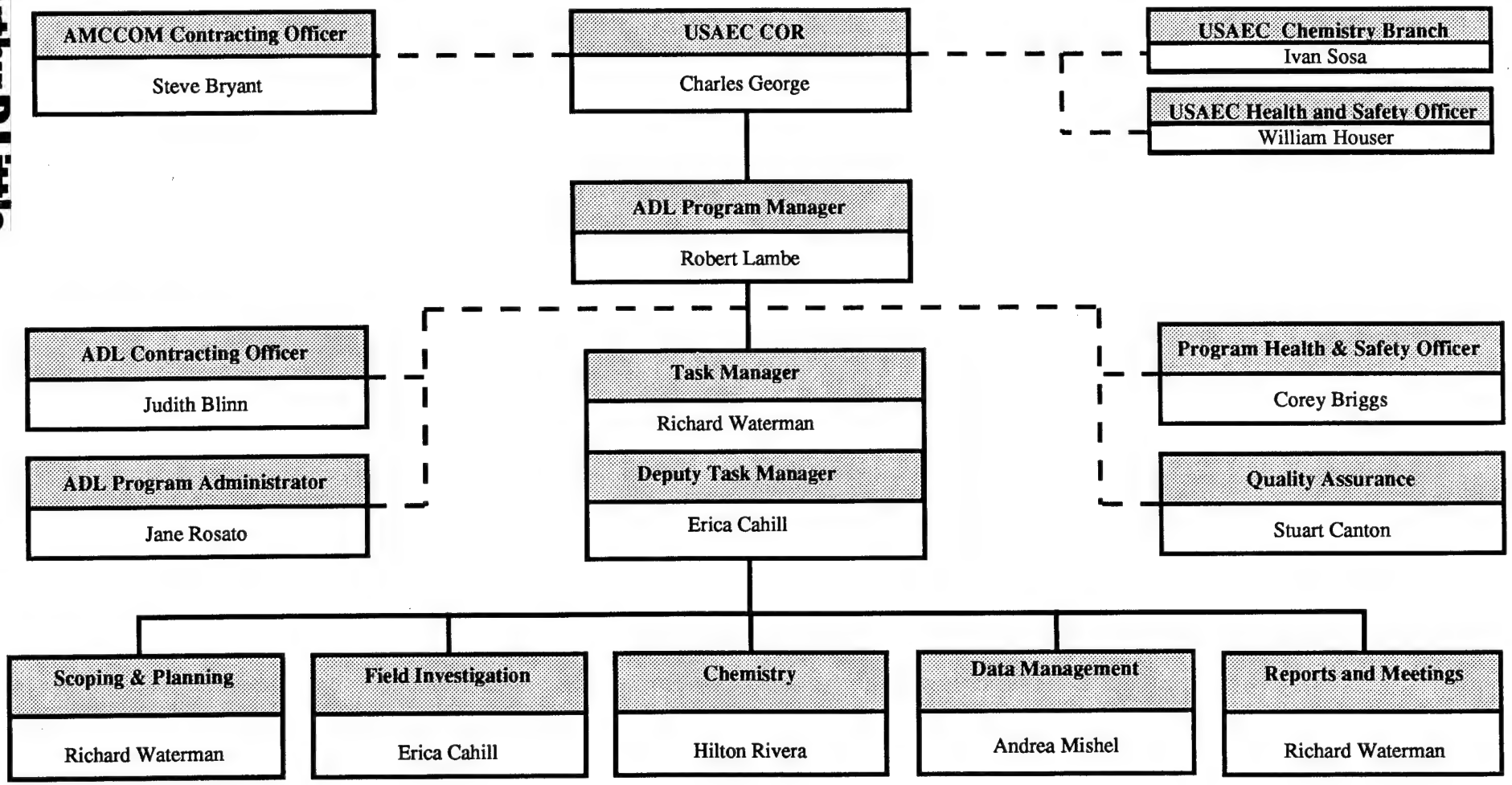
2.3 H^+GCL Project QA/QC Organization

See Appendix A.

2.4 Balsam/Dames & Moore Project QA/QC Organization

See Appendix B.

Figure 2-1C: Fort Devens Base Realignment and Closure Part II - Organizational Chart



M.devens11.Org Chart.10/94

3.0 QA Objectives for Measurement Data in Terms of Precision, Accuracy, Representativeness, Completeness, Comparability

3.1 Introduction

See Quality Assurance Project Plan.

3.2 QA Objectives for Fort Devens Data

See Appendix A and B.

3.2.1 Precision

See Quality Assurance Project Plan.

Field and analytical precision will be assessed through collection and analysis of replicate samples. One asbestos QC sample will be collected for each 20 bulk samples collected. The QC sample will be collected immediately adjacent to one of the other bulk samples. The sample will be assigned a unique sample number and will not be identified to the laboratory as a quality control sample. Results of the replicate analyses will be considered acceptable if the same type(s) of asbestos are present (or absent). If the analyses do not agree, the area will be resampled.

In addition, 10 percent of all asbestos samples are selected for reanalysis by a different analyst. Any discrepancies are resolved before reporting data to the client.

See Appendix B for precision standards of lead paint sampling program.

3.2.2 Accuracy

See Quality Assurance Project Plan.

The accuracy of asbestos analysis is routinely assessed through participation in the National Voluntary Laboratory Accreditation Program (NVLAP) administered by the National Institute of Standards Technology. This program requires routine analysis of quality control samples containing known quantities and types of asbestos.

Certain types of samples such as plasters and certain types of other surfacing materials which are known to pose PLM analysis difficulties will be reanalyzed by the QC Coordinator. An analyst may request that the QC Coordinator or Laboratory Director provide a second opinion analysis on samples for which the analyst feels the results may be inaccurate.

See Appendix B for accuracy standards of lead paint sampling program.

3.2.3 Representativeness
See Quality Assurance Project Plan.

3.2.4 Completeness
See Quality Assurance Project Plan.

3.2.5 Comparability
See Quality Assurance Project Plan.

4.0 Sample Collection

4.1 Sample Containers, Preservation, and Handling

See Work Plan.

4.1.1 Sample Containers

All asbestos samples will be placed in labeled plastic bags with twist-lock tops.

4.1.2 Sample Preservation and Holding Times

N/A

4.2 Field QC Samples

Asbestos field duplicates will be collected at a frequency of one per twenty samples.

4.3 Sample Handling

See Appendix A and B.

4.4 Sampling Equipment and Procedures

See Appendix A and B.

4.4.1 Test Pit Sampling Procedures

N/A

4.4.2 Surface Water Sampling Procedures

N/A

4.4.3 Sediment Sampling Procedures

N/A

4.4.4 Surface Soil Sampling Procedures

N/A

4.4.5 Concrete/Asphalt Chip Sampling Procedures

N/A

4.4.6 Soil Boring Procedures

N/A

4.4.6.1 Subsurface Clearance Program. N/A

4.4.7 Ground Water Sampling Procedures
N/A

4.4.8 Wipe Sampling Procedures
N/A

4.4.9 Sample Location and Elevation Survey Procedures
N/A

4.4.10 Investigation-Derived Waste Handling Procedures
N/A

4.4.11 Geoprobe Sampling
N/A

5.0 Sample Custody

5.1 Field Custody Procedures

See Appendix A and B.

5.2 Laboratory Custody Procedures

See Quality Assurance Project Plan.

After collection of an asbestos sample, all sampling information will be recorded on a three-part Asbestos Bulk Sample Analysis Record (see Draft Supplemental Work Plan).

A unique sample identification will be assigned to each sample, this number will be placed on both the record form and asbestos sampling bag. All sample transfers will be documented on a chain-of-custody form.

6.0 Calibration Procedures and Frequency

See Appendix A and B.

6.1 Field Instrumentation

N/A

6.2 Laboratory Calibration

Laboratory calibration procedures will be performed in accordance with the requirements of the analytical procedures.

6.2.1 Laboratory Instrumentation Calibration

6.2.1.1 Calibration Standards. N/A

6.2.1.2 Calibration Frequency. N/A

6.2.1.3 Tuning and GC/MS Mass Calibration. N/A

6.2.1.4 Decafluorotriphenylphosphine (DFTPP). N/A

6.2.1.5 p-Bromofluorobenzene (BFB). N/A

6.2.2 Operational Calibration

N/A

6.2.2.1 General Calibration Procedures. N/A

6.2.2.2 Method Blank. N/A

6.2.2.3 Calibration Curve. N/A

6.2.3 Calibration for USAEC Approved Methods

N/A

7.0 Analytical Procedures

See Appendix A and B.

7.1 Analytical Program

See Appendix A and B.

7.2 Laboratory Method Approval

See Appendix A and B.

7.2.1 Laboratory Methods Requiring Approval

N/A

7.2.2 Methods Not Requiring Approval

See Quality Assurance Project Plan.

7.3 Analyst Qualification

See Appendix A.

7.4 Analytical Methods

See Appendix A and B.

7.4.1 Sulfate and Chloride

N/A

7.4.2 Volatile Organics (GC/MS)

N/A

7.4.3 Semivolatile (Acid/Base/Neutral) Organics (GC/MS)

N/A

7.4.4 Organochlorine Pesticides/PCBs (GC/ECD)

N/A

7.4.5 Metals

7.4.5.1 Inductively Coupled Plasma Atomic Emission Spectroscopy (ICAP). N/A

7.4.5.2 Cold Vapor (Mercury). N/A

7.4.5.3 Graphite Furnace Atomic Absorption. N/A

7.4.6 Explosives
N/A

7.4.7 TSS (Total Suspended Solids)
N/A

7.4.8 TPHC (Total Petroleum Hydrocarbons by Infrared)
N/A

7.4.9 TOC in Sediment by IR
N/A

7.4.10 Total Phosphorous and Phosphate
N/A

7.4.11 Total Kjeldahl Nitrogen (TKN) in Water by Automated Spectrophotometry
N/A

7.4.12 Organophosphorus Pesticides
N/A

7.4.13 Chlorinated Herbicides
N/A

7.4.14 Nitrate
N/A

7.4.15 Hardness
N/A

7.4.16 Alkalinity
N/A

7.4.17 Asbestos (Bulk) by Polarizing Light Microscopy
See Appendix A.

7.4.18 Particle Size by Sieve Analysis

N/A

7.4.19 TCLP Leachate Preparation

N/A

7.5 Field Analytical Methods

N/A

8.0 Data Reduction, Validation, and Reporting

See Appendix A and B.

8.1 Arthur D. Little's Data Management

See Quality Assurance Project Plan.

A database will be used to document the results of the asbestos and lead based paint survey. Data entry will occur during the field investigation and will include information collected and included on the USAEC ACM Assessment Checklist (see Work Plan). Analytical results will be entered into the database corresponding with the appropriate building or structure. The database format will be compatible with dBaseIV.

8.1.1 Flow of Map Data into the IRDMIS
N/A

8.1.2 Flow of Geotechnical Data into the IRDMIS
N/A

8.1.3 Flow of Chemical Data into the IRDMIS
N/A

8.2 Data Reduction

See Appendix A.

8.3 Data Validation

See Appendix A and B.

8.3.1 USAEC Data Validation Procedures
N/A

8.3.2 USEPA Data Validation Procedures
N/A

8.4 IRDMIS Record and Group Checks

N/A

8.5 Data Reporting

See Appendix A.

A database will be used to document the results of the asbestos and lead based paint survey. Data entry will occur during the field investigation and will include information collected and included on the USAEC ACM Assessment Checklist (see Work Plan). Analytical results will be entered into the database corresponding with the appropriate building or structure. The database format will be compatible with dBaseIV.

9.0 Internal QC Checks and Frequency

9.1 Control Samples

See Appendix A and B.

9.2 Field Control Samples

See Appendix A and B.

9.2.1 Trip Blanks
N/A

9.2.2 Field Equipment/Rinsate Blanks
N/A

9.2.3 Field Duplicates
See Appendix A and B.

9.2.4 Field Blanks
N/A

9.3 Laboratory Control Samples

See Appendix A and B.

9.3.1 Laboratory Blanks
N/A

9.3.2 Laboratory Duplicates
N/A

9.3.3 Calibration Standards
N/A

9.3.4 Spike Sample
N/A

9.3.5 Internal Standard
N/A

9.4 Concentration and Frequency of Control Samples

See Appendix A and B.

9.4.1 Class 1 Certified Method

N/A

9.4.2 Class 1A Certified Method (GC/MS only)

N/A

9.5 Data Reporting for QC

9.5.1 Class 1, Class 1A, and Class 1B Certified Methods

N/A

10.0 Performance and System Audits

10.1 Field Audits

N/A

10.2 Laboratory Audits

See Appendix A and B.

The asbestos laboratory participates in the National Voluntary Laboratory Accreditation Program administered by the National Institute of Standards Technology.

10.2.1 Data Review

See Appendix A and B.

10.3 Project Audits

See Appendix A and B.

11.0 Preventive Maintenance

11.1 Field Instruments

See Appendix B.

11.2 Laboratory Equipment

See Appendix A and B.

12.0 Procedures Used to Assess Data Accuracy, Precision, and Completeness

12.1 Lack of Fit (LOF) and Zero Intercept (ZI) Tests

N/A

12.2 Certified Reporting Limit (CRL)

N/A

12.3 Method Certification Accuracy

N/A

12.4 Method Certification Standard Deviation

N/A

12.5 Method Certification Percent Inaccuracy

N/A

12.6 Method Certification Percent Imprecision

N/A

12.7 Data Moving-Average Accuracy and Precision

N/A

12.8 Control Charts

N/A

12.8.1 Control Chart Plotting: Single-Day

N/A

12.8.2 Three-Point Moving Average

N/A

12.9 Out-of-Control Conditions

See Appendix A.

12.10 Non-USAEC Methods

See Appendix A.

12.11 Completeness

See Quality Assurance Project Plan.

13.0 Corrective Actions

See Appendix A and B.

13.1 Field Situations

See Appendix B.

13.2 Laboratory Situations

See Appendix A.

14.0 Quality Assurance Reports to Management

14.1 Laboratory Reports

See Appendix A and B.

14.2 Program QA Officer and Lead Chemist Reports

See Quality Assurance Project Plan.

Appendix A

HGCL Analytical Services Laboratory
A Division of HGCL, Inc.

98 North Washington Street
Boston, MA 02114
(617) 589-0660

QUALITY ASSURANCE/QUALITY CONTROL AND PROCEDURES MANUAL

Written By: Bryan Clark
Adrian Stanca
Mark Fish
Jack Yee

Version 4.0
Revised 05/31/93

TABLE OF CONTENTS

POLICY STATEMENT

The primary mission of HGCL Analytical Services Laboratory (HASL) is to provide high quality analyses within reasonable turnaround times at competitive prices. The purpose of the QA/QC Manual is help assure that the measurements and data generated at HASL are precise and accurate within reasonable upper and lower control limits. All Analysts will use the procedures described in the Manual for routine analyses. Any deviation from the prescribed methodologies should be defendable in writing at the time they are performed. QA/QC is the responsibility of all personnel at HGCL and it is imperative that staff members are not subjected to undue pressure or inducement that may influence their judgement or the results of their analysis.

OVERVIEW

HASL's QA/QC Manual is organized into five parts (A-E) as described below:

Part A: General information that applies to all analytical procedures

Part B: Transmission Electron Microscopy (TEM)

Part C: Phase Contrast Microscopy (PCM)

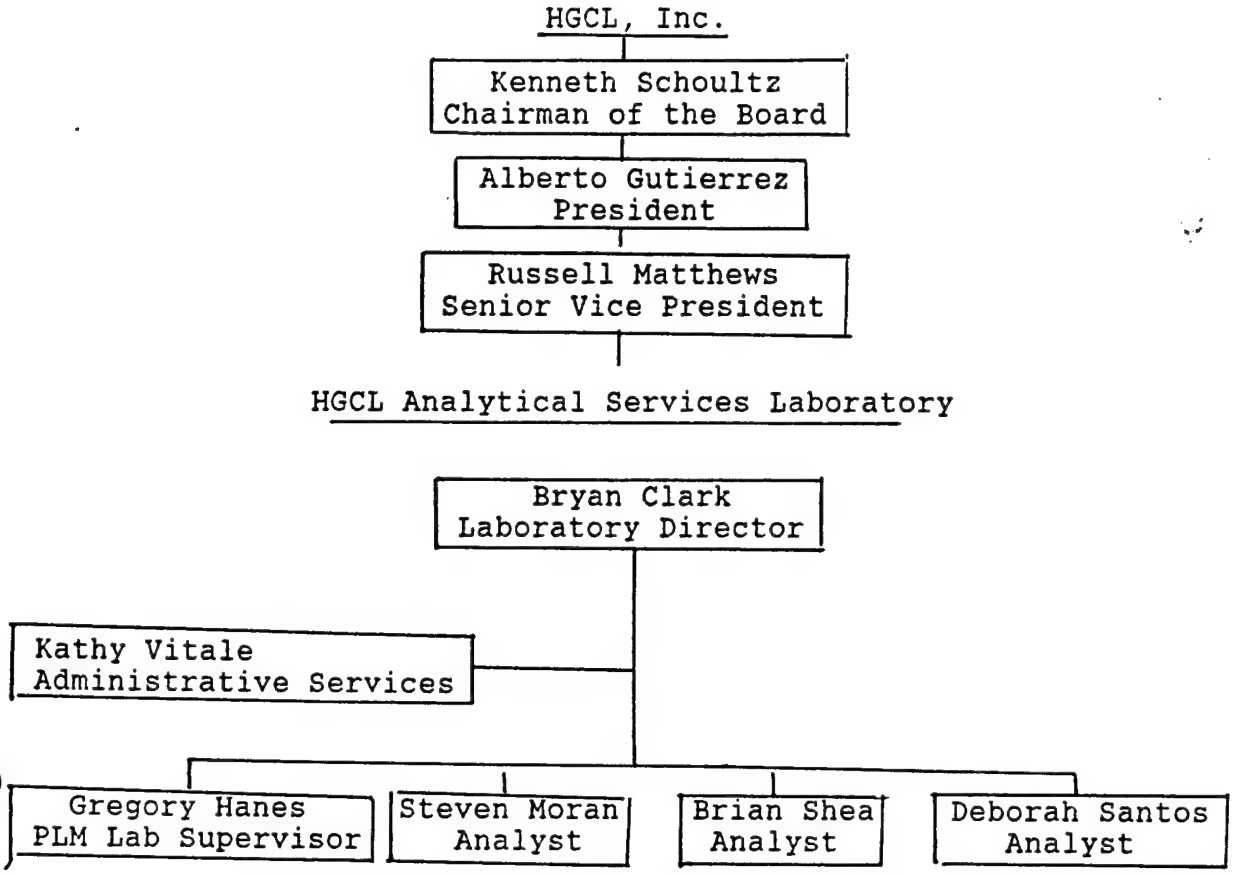
Part D: Polarized Light Microscopy (PLM)

Part E: Appendices

PART A

GENERAL INFORMATION

A.1 Laboratory Organization and Responsibility



The Laboratory Director maintains overall responsibility for the Laboratory. Administrative Services is responsible for receipt and routing of samples to the appropriate analysts. The Supervisor enforces the QC system and submits reports to the Laboratory Director on a Quarterly basis.

Condensed Staff Resumes

BRYAN CLARK

M.S. (Geology) University of Michigan
B.S. (Geology) University of Wisconsin

Over seven years in electron microscopy with more than five years in the field of asbestos analysis.

Member of the New England Society of Electron Microscopy, and the National Asbestos Council.

Successfully completed TEM course at McCrone Environmental Services, Inc. and the McCrone Research Institute's Microscopical Identification of Asbestos Course.

GREG HANES

B.S. (Geology) Tufts University

Seven years experience in field and laboratory asbestos consulting.

Member of the New England Society of the American Industrial Hygiene Association.

Successfully completed NIOSH 582 Equivalent and a course on the analysis of asbestos by TEM taught by a NVLAP Assessor.

STEPHEN MORAN

B.A. (Environmental Geology) Boston University

School-related experience in mineralogy, coastal geology and sedimentology.

Member of the New England Section of the American Industrial Hygiene Society.

Successfully completed NIOSH 582 Equivalent, McCrone Research Institute Microscopical Identification of Asbestos, and a course on the analysis of asbestos by TEM taught by a NVLAP Assessor.

BRIAN SHEA

B.S. (Geology) Salem State College

School-related field experience in mineralogy, petrology, hydrology, geomorphology and sedimentology.

Member of the New England Section on the American Industrial Hygiene Society.

Successfully completed NIOSH 582 Equivalent, HGCL's Analyst Training Program and a course on the analysis of asbestos by TEM taught by a NVLAP Assessor.

DEBORAH SANTOS

B.S. (Earth Science) University of Massachusetts

School-related experience in mineralogy, petrology and computers. Five years experience in asbestos analysis in bulks, air, water and dust utilizing PLM, PCM and TEM.

Successfully completed NIOSH 582 Equivalent and Hygeia Inc.'s Bulk Sample Analysis for Asbestos: PLM Course.

Staff Qualifications

Laboratory Director:

Primary

- Bachelors Degree (Masters Degree preferred) from a recognized college or university with a major in one of the physical sciences.
- At least two years experience in asbestos analysis.
- A thorough understanding of the principals and practices of PLM and TEM and qualified to conduct TEM and PLM analyses including the measurement and interpretation of the electron diffraction patterns and energy dispersive x-ray spectrometry, as well as measurement of refractive index using Becke lines or dispersion staining techniques.

Secondary

- Knowledge of the preparation of air filters, dust, floor tile and water samples for TEM analysis, and a variety of bulk materials for PLM analysis.
- Understands proper sample handling, preparation, analysis, storage, disposal, quality system, contamination monitoring, and control, for asbestos analysis.

Analysts:

Primary

- Bachelors degree from a recognized college or university with a major in one of the physical sciences.
- At least one year's experience as either an electron or optical microscopist. College course work can be counted towards this requirement.

Secondary

- Knowledge of crystallography and mineralogy.
- Knowledge of computer data entry and software utilization.
- Ability to work with hands on microscopic materials utilizing tweezers and scalpels.

Administrative Services:

Primary

- High school diploma.
- Ability to operate existing computer programs to reduce, compile and store analytical data and write reports.
- Organizational skills to keep track of samples to be analyzed as well as those already archived.

Secondary

- Flexibility to help out in other areas as needed.
- Ability to interface with the branch offices of HGCL.

STAFF ASSIGNMENTS

Laboratory Director:

Maintains the quality system and associated quality manual. Talks with clients and other HGCL personnel and management about questions regarding laboratory operation and practices. Schedules appropriate laboratory personnel to specific duties and times. Maintains up-to-date knowledge of asbestos analysis by associating with peers and fellow microscopist through conferences, meetings and personal interaction. Maintains laboratory budget and accreditations, and oversees that all supplies and equipment are available for analyst's use.

Senior Laboratory Analyst:

Operates analytical transmission electron microscope and/or Polarized Light Microscope performs routine asbestos analysis. Performs minor TEM and/or PLM maintenance and keeps microscope aligned. Preps and analyzes samples in accordance with the provisions of this manual. Informs laboratory director of needed equipment and/or supplies. Informs laboratory director of any malfunctioning equipment.

Laboratory Clerk:

Logs in all samples, checks for chain of custody forms. Makes sure arriving packages are in good shape, cleans cassettes, logs out all samples. Assigns sample status as priority or nonpriority. Makes laboratory report for review by laboratory director before release of information.

A.2 Equipment List

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Philips TEM (EM300) TEM Room #1	D 1215	07/89	03/93
Philips TEM (EM300) TEM Room #2	D 1227	01/90	03/93
EDAX X-Ray Analyzer (PV9900) TEM Room #1	HX 10767/01	04/87	01/93
EDAX X-Ray Analyzer (PV9160) TEM Room #2	HX 622/02	04/90	~~08/91~~ 07/93
Kinetic Systems Vibration Mount (308026-04-0054) TEM Room #1	121-249-2	12/90	02/93
Kinetic Systems Vibration Mount (320502-01-0075) TEM Room #1	121-249	12/90	02/93
Kinetic Systems Vibration Mount (308028-02-0054) TEM Room #1	121-249-3	12/90	02/93
Kinetic Systems Vibration Mount (308026-03-0054) TEM Room #1	121-249-1	12/90	02/93
Kinetic Systems Vibration Mount (308026-03-0054) TEM Room #2	121-250-1	12/90	02/93
Kinetic Systems Vibration Mount (308028-02-0054) TEM Room #2	121-250-3	12/90	02/93
Kinetic Systems Vibration Mount (308502-01-0705) TEM Room #2	121-250	12/90	02/93
Kinetic Systems Vibration Mount (308026-04-0054) TEM Room #2	121-250-2	12/90	02/93

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Kawanee Fume Hood (S6268608) TEM Sample Prep Area	N/A	01/90	12/92
Envirco Fume Hood (EACI10577) Sample Reception Area	87074561	09/85	12/92
Labconco Class 100 HEPA Hood (80826-01) TEM Sample Prep Area	N/A	01/90	12/92
McCrone Neg. Pres. HEPA Hood N/A PLM Station #1	45310590	01/90	.....
McCrone Neg. Pres. HEPA Hood N/A PLM Station #3	1070389	01/90	.....
McCrone Neg. Pres. HEPA Hood N/A PLM Station #4	1068389	01/90	.....
McCrone Neg. Pres. HEPA Hood N/A PLM Station #5	1069389	01/90	.....
Customized Neg. Pres. HEPA Hood N/A PLM Station #2	N/A	04/86	.....
Customized Neg. Pres. HEPA Hood N/A PLM Station #6	N/A	04/86	.....
Nilfisk HEPA Vacuum GSD 115 PLM Station #1	2115180	01/90	04/93
Nilfisk HEPA Vacuum GSD 115 PLM Station #2	1938279	04/88	04/93
Nilfisk HEPA Vacuum GSD 115 PLM Station #3	2115222	01/90	04/93

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Nilfisk HEPA Vacuum GSD 115 PLM Station #4	1910171	04/88	04/93
Nilfisk HEPA Vacuum GSD 115 PLM Station #5	1947911	04/88	04/93
Nilfisk HEPA Vacuum GSD 115 PLM Station #6	2115222	01/90	04/93
Nilfisk HEPA Vacuum GSD 115 ~~Sample Storage Room~~ → TEM Prep Room	1791167	04/86	04/93
Nilfisk HEPA Vacuum GSD 115 Sample Storage Room	1823685	04/86	04/93
Olympus PLM Microscope (BH2) PLM Station #1	205927	04/88	.....
Olympus PLM Microscope (BH2) ~~PLM Station #2~~ TEM Prep Area	211724	04/90	.....
Olympus PLM Microscope (BH2) PLM Station #3	208845	04/86	.....
Olympus PLM Microscope (BH2) PLM Station #4	211714	04/88	.....
Olympus PLM Microscope (BH2) PLM Station #5	204934	04/86	.....
Olympus PLM Microscope (BH2) PLM Station #6	206045	04/88	.....
Olympus PLM Microscope (BH2) PLM Station #7	211757	04/88	.....

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Olympus Stereoscope (SZIII) TEM Prep Area	336026	04/86	.....
Olympus Stereoscope (SZIII) PLM Station #1	336064	04/86	.....
Olympus Stereoscope (SZIII) PLM Station #2	335839	04/90	.....
Olympus Stereoscope (SZIII) PLM Station #3	336378	04/86	.....
Olympus Stereoscope (SZIII) PLM Station #4	336027	04/86	.....
Olympus Stereoscope (SZIII) PLM Station #5	296172	04/86	.....
Olympus Stereoscope (SZIII) PLM Station #6	332398	04/86	.....
Dollan & Jenner Fiber Optic Light (180) TEM Sample Prep Area	N/A	01/90	.....
Dollan & Jenner Fiber Optic Light (180) PLM Station #1	N/A	04/86	.....
Dollan & Jenner Fiber Optic Light (180) PLM Station #3	N/A	04/86	.....
Dollan & Jenner Fiber Optic Light (180) PLM Station #4	N/A	04/86	.....
Dollan & Jenner Fiber Optic Light (180) PLM Station #5	N/A	04/86	.....

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Dollan & Jenner Fiber Optic Light (180) PLM Station #6	N/A	04/86	.....
Olympus Light Source (TL 2) PLM Station #2	N/A	04/86	.....
Hitachi Video Camera PLM Station #7		01/90	.....
Sony High Resolution Color Monitor PLM Station #7		01/90	.....
Olympus PCM Microscope (BH2) PCM Room	204680	01/90	.....
HP 386 Hard Drive (Vectra RS/25C) Laboratory Director's Office	3029A09231	05/90	.....
IDS 8088 Hard Drive (PC88) Spare Office	H79002990	04/87	.....
Emerald 386 Hard Drive (240-11100) TEM Log In Area	24068711	04/87	08/92
IDS 286 Hard Drive (PC286T) Sample Reception Area	H78001386	05/88	.....
HP Monochrome Monitor (D1181G) Laboratory Director's Office	2919Y08391	05/90	.....
IBM Monochrome Monitor (5151001) Spare Office	N/A	04/87	.....
Mitsubishi Monochrome Monitor (XC-1409C) TEM Log In Area	4228926	04/87	.....
upgraded to 386 Supreme 286 Hard Drive (Standard 286) Offic Area	# 88073042	06/93	

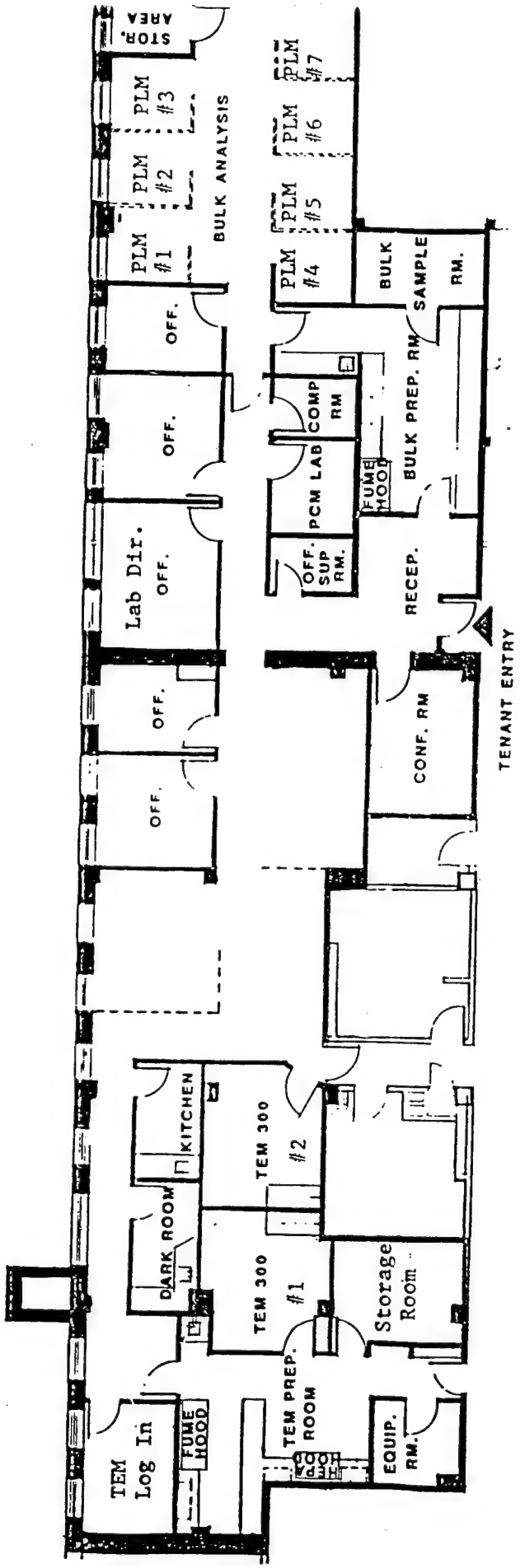
Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Magnavox Monochrome Monitor (7BM623-074G) Sample Reception Area	41243347	05/88	.....
Epson Printer (LQ-2550) Laboratory Director's Office	OHA10017199	05/90	.....
Epson Printer (RX 80F/T) Spare Office	059039	04/87	.....
Epson Printer (LQ 1050) Sample Reception Area	OTG1000020	05/88	.....
HP Printer (2225D) TEM Room #1	2916542189	04/87	.....
Digital Printer (LA 50) TEM Room #2	0126794A	04/90	.....
Hitachi FAX Machine (HF 35E) Office Area	0788	04/86	.....
Ricoh Copy Machine (FT 2260) Office Area	AM664	01/90	03/93
Mettler Analytical Balance (AJ100) Sample Reception Area	111216	10/89	02/93
Arkay Film Drier (CD20) Darkroom	D-173	01/90	.....
Arkay Print Drier (RC-1100) Darkroom	1-2444D	01/90	.....
Bessler Englarger (45V-XL) Darkroom	N/A	01/90	.....
HP Laserjet Printer (HP 2686A) Sample Office Area	# 2531 J43115	06/93	

~~File~~

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
PELCO Film Developer (26312) Darkroom	21089-2	01/90	.....
Denton Vacuum Evaporator (DV 515) TEM Sample Prep Area	655	02/93	01/93 03/93
SPI Plasma Asher (11005) TEM Sample Prep Area	1574	10/88	02/93
Haskris Water Chiller (R075) Chiller Room	H-4056	04/87	02/93
IBM Typewriter (6783) General Office Area	11-0290501	01/90	.....
Pitney Bowes Postage Meter (6200) General Office Area	72735	01/90	.....
Pitney Bowes Postage Scale (5820) General Office Area	93375	01/90	.....
Branson Ultrasonic Bath (1200) TEM Sample Prep Area	1200 R/4	07/87	.....
Thermolyne Hot Plate (HP18325) TEM Sample Prep Area	30518730	01/90	.....
Theremolyne Hot Plate (2300) PLM Station #1	37112	04/86	.....
Thermolyne Stir Plate (S18525) Darkroom	30721454	01/90	.....
Corning Stir Plate (PC 353) Darkroom	N/A	04/86	.....

Equipment (Model) Location	Serial #	Date Placed in Service	Most Recent Service
Dawson Hi-Vol Pump (1990) TEM Sample Prep Area	0493	04/93	.....
Emerson Hi-Vol Pump (SA55NXGTE-4870) TEM Sample Prep Area	0589	01/90	.....
Thermolyne Muffle Furnace (F47900) Sample Reception Area	479010406	05/87	.....
✓ Ditto		07/93	

A.3 Laboratory Layout



A.4 Chain of Custody

The chain of custody originates in the field at the time of sample collection. The Field Technician taking the sample records all pertinent information on the sample and on the data sheet. He/she then signs and dates the data sheet which now acts as chain of custody for the samples. This sheet should accompany the samples until their disposition. Each person involved with those samples should sign the chain of custody each time the samples change hands. Samples that are shipped by commercial carriers are not subject to these chain of custody requirements.

When bulk samples arrive in the laboratory, they are logged into the system by Administrative Services. At this time, the chain of custody is signed and the samples are assigned a unique laboratory ID number, checked for their integrity, sorted and labelled. The information supplied by the Field Technician should match with any information written on the individual samples. Bulk samples are then placed in the sample bins according to priority along with their data sheet/chain of custody forms. The analyst will fill out the chain of custody information when he/she analyzes the samples.

When TEM samples arrive in the laboratory, they are assigned to a particular analyst who logs them into the system, analyzes the samples and generates the report. All chain of custody information is checked and filled in as needed.

The original data sheets are permanent records of the laboratory and will be on file for a period no less than seven years. After analysis, samples are kept for a period of no less than 90 days. If the client requests that the samples be returned to them or maintained for a longer period of time, the chain of custody is filled out to reflect that request and the samples are returned or sent to SafeSite for storage. PLM samples to be disposed of are double wrapped in 6 mil polyethylene and picked up by a certified hazardous waste contractor. Proper documentation of any sample disposal will be maintained. Electron microscope grids are retained for a period no less than 3 years.

All data associated with samples analyzed at HASL are kept on computer and protected from compromise by means of a backup storage tape. The backup tape shall be stored in aa area separate from the computer and primary storage media.

A.5 Sample Log-in and Log-out Procedure

Sample Log In

The samples are received by the administrative services. He/she must check the condition of the package on receipt. If the package is damaged, the laboratory director must be immediately notified before the package is accepted. During the log-in procedure the following items must be checked by the person who is logging in the samples:

- Field data sheet must be correctly and completely filled out.
- Chain of custody form must be properly signed and dated.
- The correct number of samples must be present with field I.D. numbers attached that match the field I.D. numbers on the data sheet.
- Bulk samples must not be sent with air samples.
- No open cassettes or missing filter plugs.

If there are any discrepancies, all information is handed over to the laboratory director as he/she can get in touch with the client or field staff to take appropriate actions.

Appropriate actions can include, but are not limited to, changing the data provided on the field sheet with the clients or field staffs permission; documentation of damaged cassettes and/or shipping carton, and documentation of missing plugs and cassette tops.

Samples are then logged into the computer. If the samples are TEM air samples, count sheets will be generated. These count sheets should accompany the samples when they are turned over to the analyst.

Sample Log Out

Upon completion of analysis, the analyst or administrative services will log the samples out of the computer and a report will be generated.

If the samples are a rush, the client should be notified by Fax or verbally by phone.

A.6 Record Keeping

Records of Maintenance and Calibration

These records will be kept for each major piece of equipment. The information recorded will include the date and type of maintenance or calibration performed as well as the date of next scheduled maintenance or calibration.

Test Reports

Test reports should include the following information:

1. Name and address of the Laboratory
2. Identification of the test report by job number, batch number and date
3. Name and address of client
4. Identification and/or general description of test performed
5. Description of sampling procedure if appropriate
6. Any deviations from the test method used
7. Measurements, examinations and derived results
8. A statement of measurement uncertainty when appropriate
9. A statement that the results may not be reproduced without the permission of the laboratory
10. A statement that the test report relates only to the items tested

Personnel Records

Personnel records of the laboratory staff will contain the following information:

1. Education, training and technical knowledge or experience that qualifies the individual for his/her assigned functions
2. Job description including their assignment and responsibilities
3. A description of any in-house training they have received
4. Results of any tests they have been given for competence in their assigned duties (eg. duplicates, replicates, proficiencies)
5. A signed statement saying the individual has read and understands the QA/QC Manual

A.7 Training

Each analyst is prepared for the tests he/she is responsible for performing. Training consists of two units: 1) formal training provided by an approved course (eg McCrone) and/or in-house training and 2) individual training conducted by a senior staff member as needed.

The level of competency of each analyst is tested frequently through duplicate, replicate and inter-laboratory analyses, as well as proficiencies from NIST, NIOSH and various states in which HASL is certified.

The following training outlines for PLM and TEM Analysts are used for in-house training. A new employee with no previous training would be required to spend a full 40 hours training under an experienced microscopist learning the concepts and techniques outlined. A new employee with previous experience may only require a fraction of this training. In that case, the outline could be tailored to suit his or her background and experience level.

PLM Training Outline

I. Introduction to Asbestos and Its Application

II. Optical Mineralogy/Crystallography
  A. Optical Parameters
    1. Morphology
    2. Color and Pleochroism
    3. Refractive Index - Parallel and Perpendicular
      a. Dispersion Staining
      b. Becke Line
    4. Extinction Characteristics
    5. Sign of Elongation
    6. Birefringence
  B. Laboratory Practical

III. Dispersion Staining Curve Generation and Evaluation

IV. Anatomy of a Microscope

V. Issues
  A. Health Effects
  B. Abatement
  C. Lab Safety

VI. Sample Management
  A. Log-in
  B. Log-out
  C. Filing System

VII. Laboratory Documentation
  A. Log-in Sheet
  B. Quality Control
    1. QC Book
    2. Recognizing Deficiencies
  C. Computer
  D. Client Copies

VIII. Sample Preparation Techniques

IX. Practical
  A. 100% QC Analyses
  B. Identification Flow Chart
  C. Recognition of Asbestos at Stereoscope
  D. Standards and Known Materials
  E. Percentage Estimation

X. Microscope Maintenance

XI. Advanced Training/Special Topics

TEM Training Outline

I. TEM Imaging
   - A. Why TEM is necessary
   - B. Instrument Requirements
   - C. Resolution, Imaging and Contrast
   - D. Specimen-Beam Interaction
   - E. Practical Exercises Using the TEM

II. Crystallography and Diffraction
   - A. Crystal Systems
   - B. Definitions and Nomenclature
   - C. Electrons as Waves and Particles
   - D. Image Formation Theory
   - E. Miller Indices
   - F. Bragg's Law
   - G. Ewald Construction
   - H. Reciprocal Space
   - I. Camera Constant and Magnification Calibration
   - J. Indexing and Interpreting Electron Diffraction Patterns
   - K. Practical Exercises in Photography and Indexing

III. Microanalysis
   - A. Electron-excited X-ray Emission
   - B. Other Sources of X-rays
   - C. Spectrum Manipulation and Comparison
   - D. Preparing a Standards Library
   - E. Interpretation of X-ray Spectra
   - F. Practical Exercises in EDXA

IV. Sample Preparation
   - A. MCE Filters
   - B. PC Filters
   - C. Water, Dust, NOB, Quick Prep Procedures
   - D. Practical Exercises

V. Counting Rules and Data Reduction
   - A. Counting Procedures
   - B. Structure Classification
   - C. Stopping Rules
   - D. The Z Test
   - E. Data Reduction and Analysis
   - F. Practical Exercises

VI. Reporting Requirements (AHERA and ELAP)

VII. QA/QC in the TEM Laboratory
   - A. Definitions
   - B. Statistics
   - C. Blanks
   - D. Verified Counting
   - E. Duplicate/Replicate Counting
   - F. Records
   - G. QA/QC Manual

A.8 Quality Assurance Reports

Communication between analysts and the QA/QC Coordinator and the Laboratory Director is essential to the overall QA/QC program. Significant problems must be brought to the attention of the appropriate person so that corrective action, if required, can be taken before problems become unmanageable.

Quality Assurance reports shall be submitted to the Laboratory Director on a quarterly basis. The reports will include the results of audits, problems and recommended solutions.

A.9 Corrective Action

When any deficiencies under the QA plan are detected, immediate action will be taken. Deficiencies in data collection or manipulation that may require corrective actions are listed below. The corrective action is shown under the same letter label.

Deficiencies

A. Disparate duplicate or replicate counts. (Outside the accepted limits as defined in this document)
B. Mislabeled samples
C. Incorrectly calculated results
D. Improperly loaded cassette
E. Damaged cassette
F. Break in chain-of-custody
G. Malfunctioning Instrument
H. Improper log entries
I. Lost or misplaced samples
J. Bulk samples too large or too small or otherwise unusual

Corrective Actions

A. Check for systematic error, instrument malfunction, data reduction error - reconcile
B. Reject samples
C. Recalculate - If results have already been reported, notify client of altered results
D. Reject sample
E. Reject sample
F. Trace to break or reject sample
G. Repair, service or call service contractor
H. Correct entry and redefine correct entering procedure for responsible person(s).
I. Report to client or request samples to be recollected.
J. Present to Laboratory manager for appropriate action.

Include items under B,D,E,F, G, and I in QA report to Laboratory Director.

A.10 Client Complaints

Response to Testing Complaints

Complaints:

1. All complaints about the results of tests or questions about testing procedures will be submitted to the laboratory director.

2. Explanation of test results may be made by telephone or in writing. When made by telephone the essence of the conversation will be collected in a written note and included with the results of the test; a copy will also be included with QA/QC report for the test methods used.

3. Responses to complaints will be made in a reasonable time so that the actions of the client derived from any reports of tests can be effected in a timely manner.

4. All complaints must be acknowledged and every effort will be implemented to answer all reasonable questions.

PART B

TRANSMISSION ELECTRON MICROSCOPY

B.1 DEFINITION OF TERMS

Analytical Sensitivity: Airborne asbestos concentrations which are represented by each asbestos structure counted for a specific volume of air and for the proportion of the filter examined. The analytical sensitivity shall be less than 0.005 structures/cc.

Asbestiform: A specific type of mineral fibrosity in which the fibers have high tensile and flexural strength.

Aspect Ratio: The ratio of length to width of a fiber. Only fibers with an aspect ratio of 5 to 1 or greater will be counted for AHERA samples.

Bundle: Three or more fibers in a parallel configuration with interfiber spacing less than one fiber diameter.

Clean Area: An area having HEPA filtered air under positive pressure. An open laboratory blank in this area should show an average of less than 18 structures/mm2 in an area of 0.057 mm2 and a maximum of 53 structures/mm2 for any single preparation. The average will be based on two filters which are placed at the preparation site. Two grids from each filter must be examined, and each examination will include five grid openings. The total number of openings will be no less than twenty.

Cluster: A collection of fibers in a random arrangement with no single fiber isolated from the rest. These collections must have more than two intersections.

EDXA: Energy Dispersive X-Ray Analysis.

Fiber: A structure with parallel sides equal to or greater than 0.5 um in length with an aspect ratio of at least 5 to 1.

Filter Background Level: Concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank. This level is defined as 70 structures per square millimeter.

Grid: A matrix of openings similar to a window screen used as an aid in TEM examination. Grids used for TEM analyses will be 200 mesh copper approximately 3mm in diameter.

Matrix: A structure containing one or more fibers which are adhering to or are partially encapsuled by a nonasbestos particle. At least one of the exposed the fiber(s) must meet the fiber definition.

NSD: No structures detected.

SAED: Selected Area Electron Diffraction.

TEM: Transmission Electron Microscope.

B.2 TEM LABORATORY FUNCTION AND CONTROL

Function:

The use of TEM for the analysis of asbestiform minerals derives from a wide range of capabilities of analytical electron microscopes to operate at levels of detection unattainable by other methods. The combination of morphological, crystallographic and chemical information obtained from a small particles allows for their identification.

Control:

Since the TEM is a critical part of the analytical sequence, it must be controlled in a manner that insures optimum performance of both the instrument and the operator. The use of the instrument must be confined to samples described in this manual. Materials that may introduce spurious data should not be introduced into the TEM environment. Control of access should be limited to the laboratory manager and designees and the rules of operation and maintenance should be strictly enforced.

Individual operators should be thoroughly trained and should operate under the supervision of an experienced microscopist until they are comfortable with the use of all equipment associated with the TEM.

B.3 Sampling Procedure and Documentation

Introduction:

Field sampling must comply with EPA prescribed procedures. All aspects of sampling, including control measures will be fully documented. The objective of these procedures are to insure that all aspects of the sampling process are clearly understood and that the materials and equipment used in the process are properly calibrated so that final reports will be statistically meaningful.

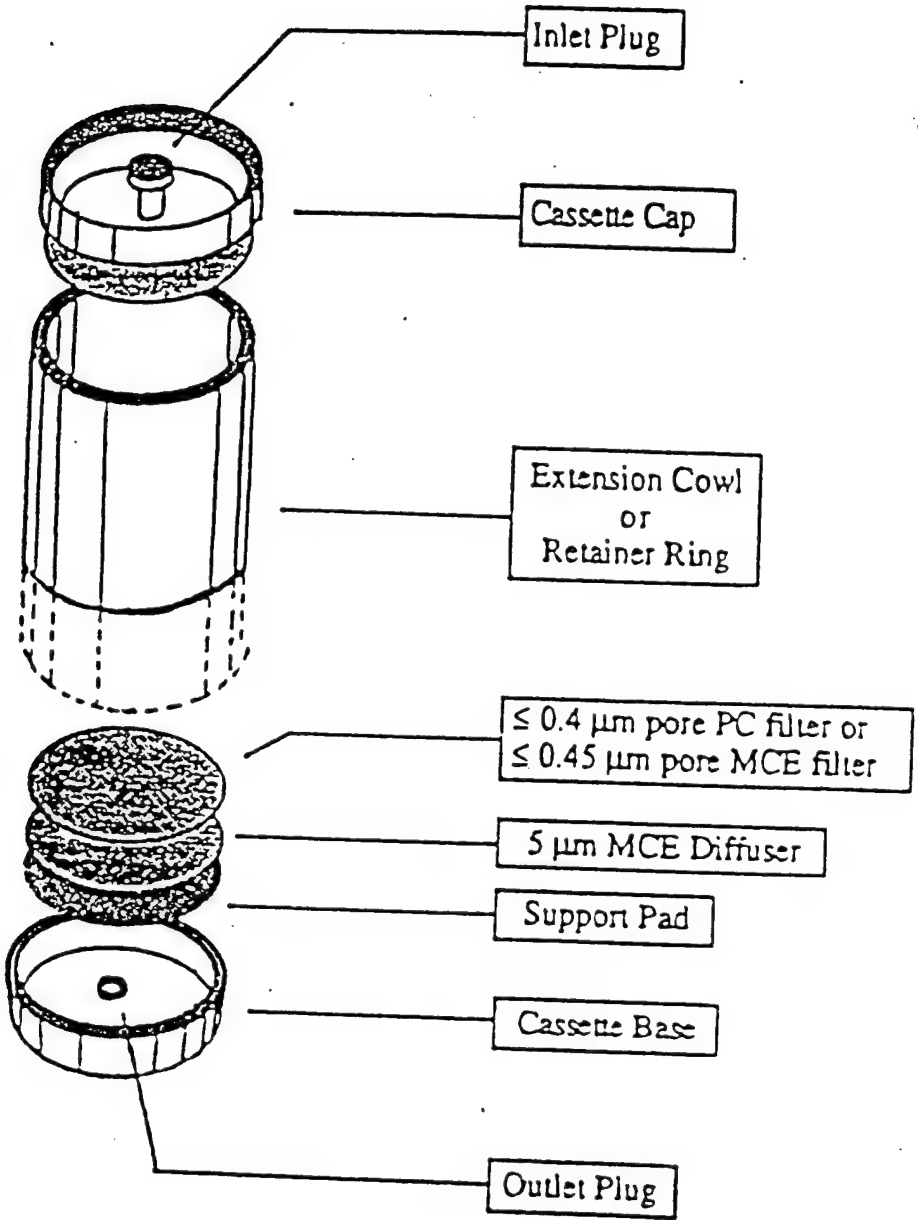
All sampling will be performed by qualified individuals as designated by the project director. Sampling will use either commercially available cassettes and collection filters, or ones prepared by HASL.

Loading cassettes and field sampling, including leak tests should be performed in accordance with EPA 40 CFR Part 763. Chain-of-custody chart should accompany all air samples.

Pumps used to sample the atmosphere will be tested for flow rate using a recognized standard. The test should be performed using a loaded cassette of the same type and kind used at the sampling site.

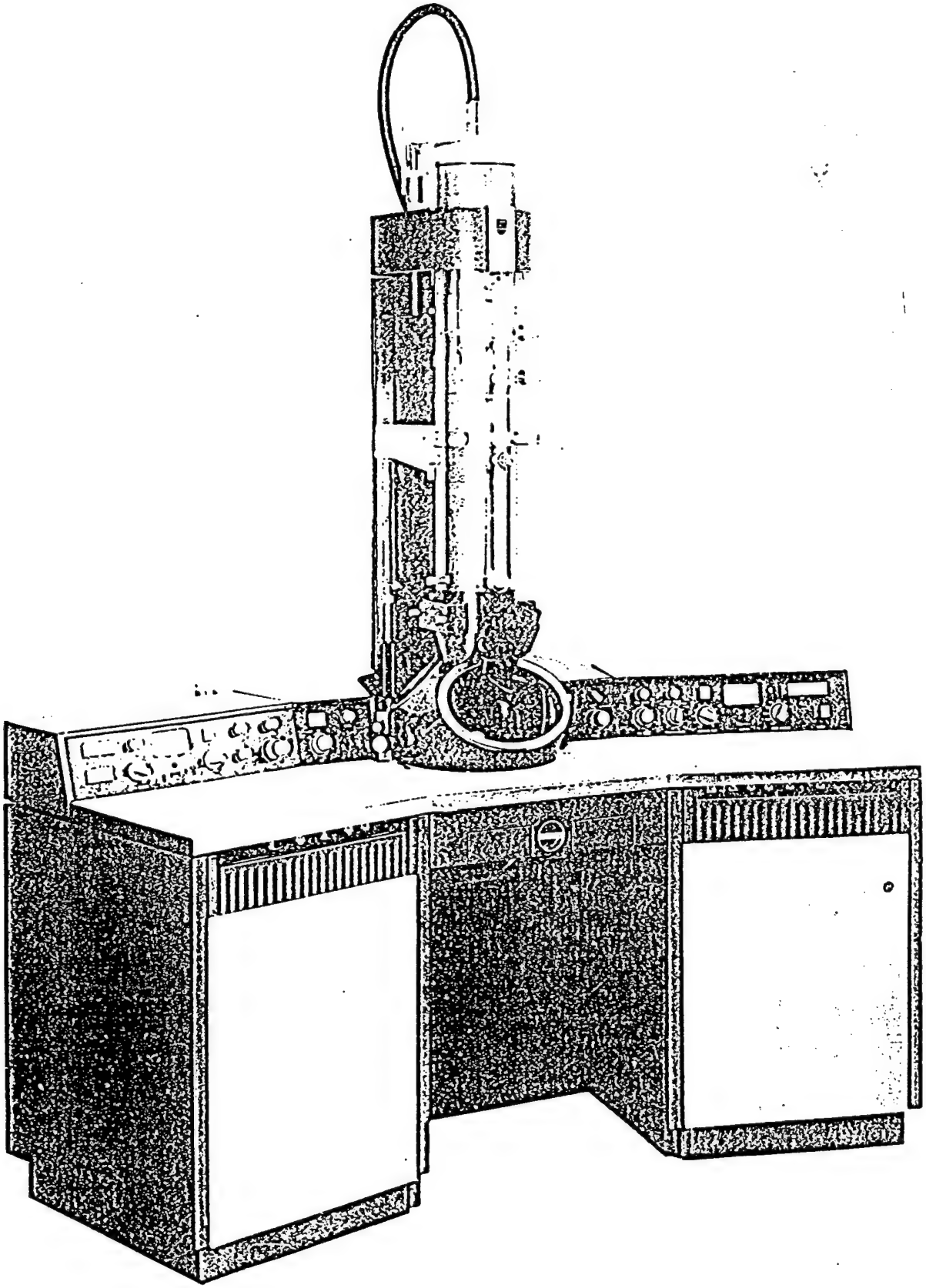
Samples collected in the field must be secured in a safe place inaccessible to unauthorized persons. Samples, together with their data sheets must be sent by the safest and quickest route. This could be hand carried or Express Mailed. Never transport, store or otherwise mingle bulk and air samples. The means of transportation and date shall be recorded on the data sheet accompanying the sample.

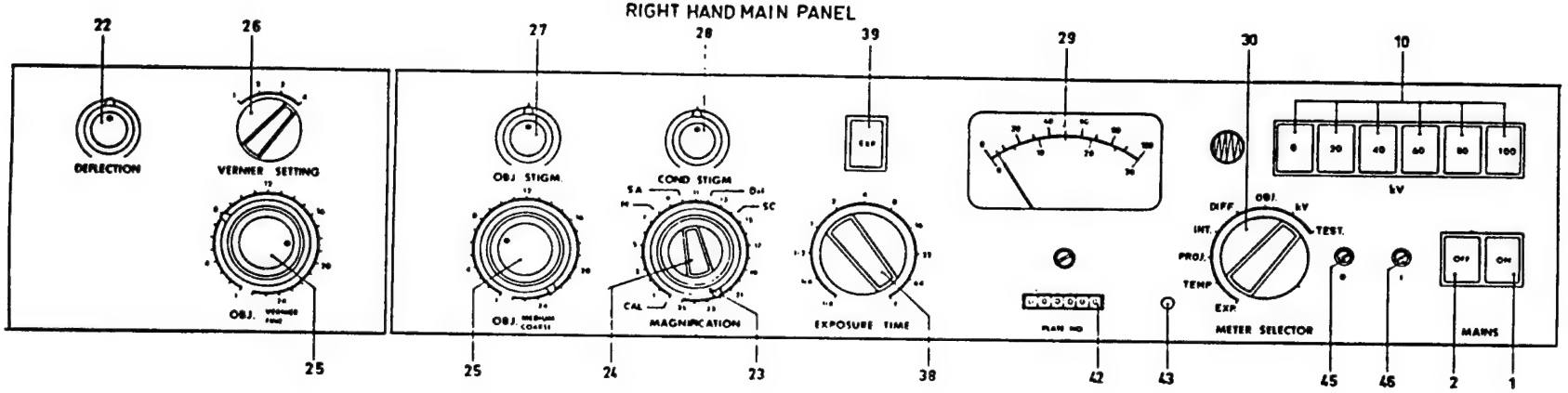
Figure 1 - Sampling Cassette Configuration



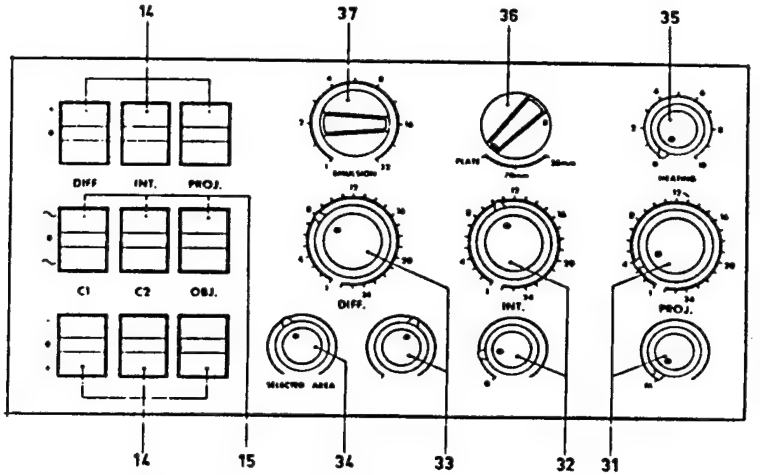
B.4 Standard Operating Procedure - Philips EM 300

Figure 2 - Philips EM300



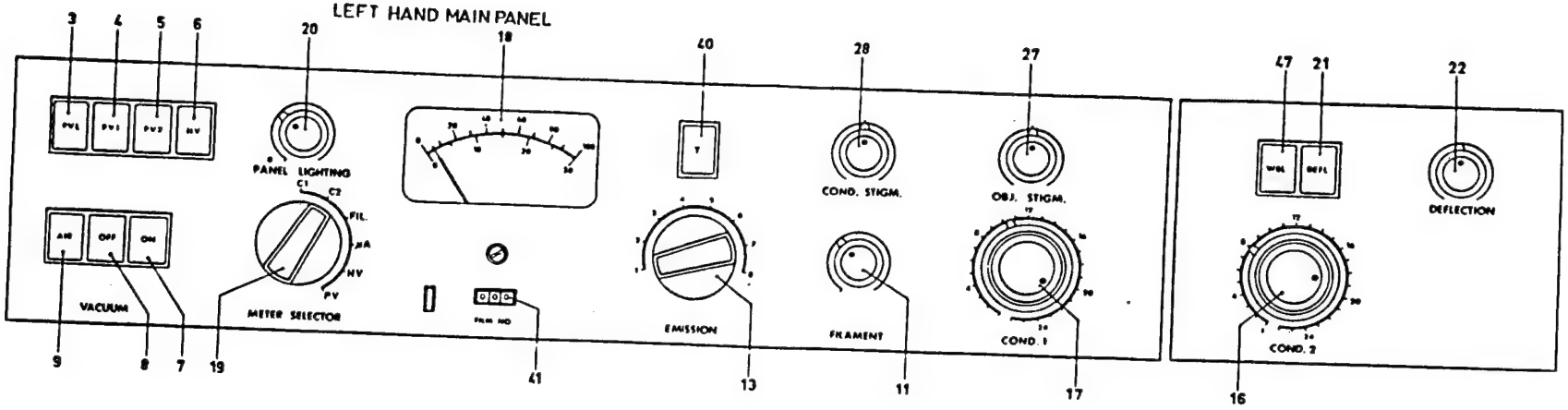


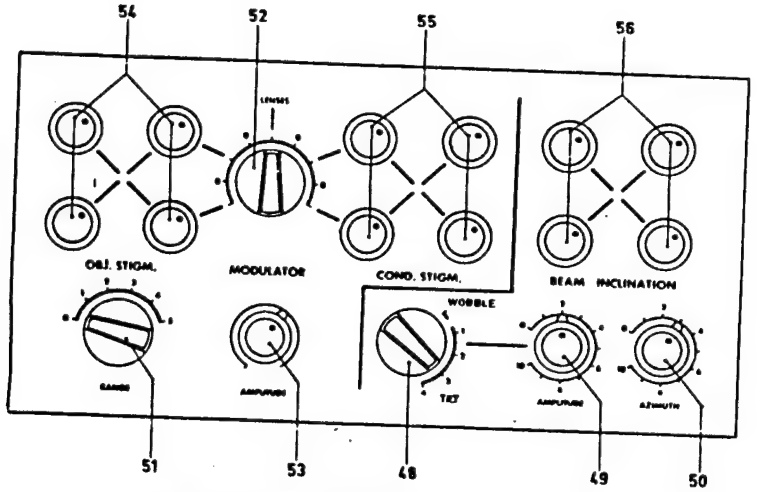
33 DIFFRACTION LENS COARS (steps) AND FINE (continuous) CONTROLS.
34. SELECTED AREA CONTROL.
35. HEATING CONTROL.
36. CAMERA SELECTOR.
37. EMULSION SELECTOR.
38. EXPOSURE TIME SELECTOR.
39. SHUTTER.
40. TRANSPORT OF CAMERAS (plate /70mm).
41. 70mm FILM COUNTER.
42. PLATE COUNTER.
43. RESET FOR PLATE COUNTER.
44. PLATE COUNTER BLOCKING. (see fig.126 no.147)
45. DIFFRACTION LENS CURRENT CALIBRATION.
46. INTERMEDIATE LENS CURRENT CAUBRATION.
47. WOBBLER ON-OFF.
48. WOBBLER/BEAMTILT QUADRANT SELECTOR.
49. BEAMTILT AMPLITUDE.
50. BEAMTILT AZIMUTH.
51. OBJECTIVE STIGMATOR RANGE.
52. MODULATOR SELECTOR.
53. MODULATOR AMPLITUDE.
54. OBJECTIVE STIGMATOR CENTRING CONTROLS.
55. CONDENSER STIGMATOR CENTRING CONTROLS.
56. BEAMTILT CENTRING CONTROLS.
57. STAND BY SWITCH.
58. LENS SUPPLY ON OFF.
59. HOUR COUNTER „TOTAL".
60. HOUR COUNTER „H.T."
61. FUSES.



RIGHT HAND AUXILLARY PANEL

Figure 3 - Electrical Control Panel Description





LEFT HAND AUXILLARY PANEL

1. MAINS ON.
2. MAINS OFF.
3. PVL - PREVACUUM LEADS.
4. PV1 - PREVACUUM PUMPING SYSTEM.
5. PV2 - PREVACUUM MICROSCOPE COLUMN.
6. HV - HIGH VACUUM MICROSCOPE COLUMN.
7. VACUUM SYSTEM ON
8. VACUUM SYSTEM OFF
9. AIR INLET.
10. HIGH TENSION SELECTORS, AND ON-OFF SWITCH.
11. FILAMENT HEATING CONTROL.
12. FILAMENT SUPPLY VOLTAGE RANGE SWITCH.
13. EMISSION SELECTOR.
14. LENS SWITCHES ON-OFF AND REVERSEL.
15. LENS MODULATOR SWITCHES.
16. CONDENSER LENS 2 COARSE (steps) AND FINE (continuous) CONTROLS.
17. CONDENSER LENS1 COARSE (steps) AND FINE (continuous) CONTROLS.
18. METER.
19. METER SELECTOR.
20. PANEL LIGHTING.
21. DEFLECTION SYSTEM ON-OFF.
22. DEFLECTION SYSTEM CONTROLS.
27. MAGNIFICATION SWITCH.
24. MAGNIFICATION FUNCTION SELECTOR.
25. OBJECTIVE LENS CURRENT CONTROLS (coarse; medium; fine; vernier)
26. OBJECTIVE LENS VERNIER SETTING.
27. OBJECTIVE STIGMATOR CONTROLS.
28. CONDENSER STIGMATOR CONTROLS.
29. METER.
30. METER SELECTOR.
31. PROJECTOR LENS COARSE (steps) AND FINE (continuous) CONTROLS.
32. INTEMEDIATE LENS COARSE (steps) AND FINE (continuous) CONTROLS.

Figure 4 - Electrical Control Panel Description

Figure 5 - Microscope Column Description

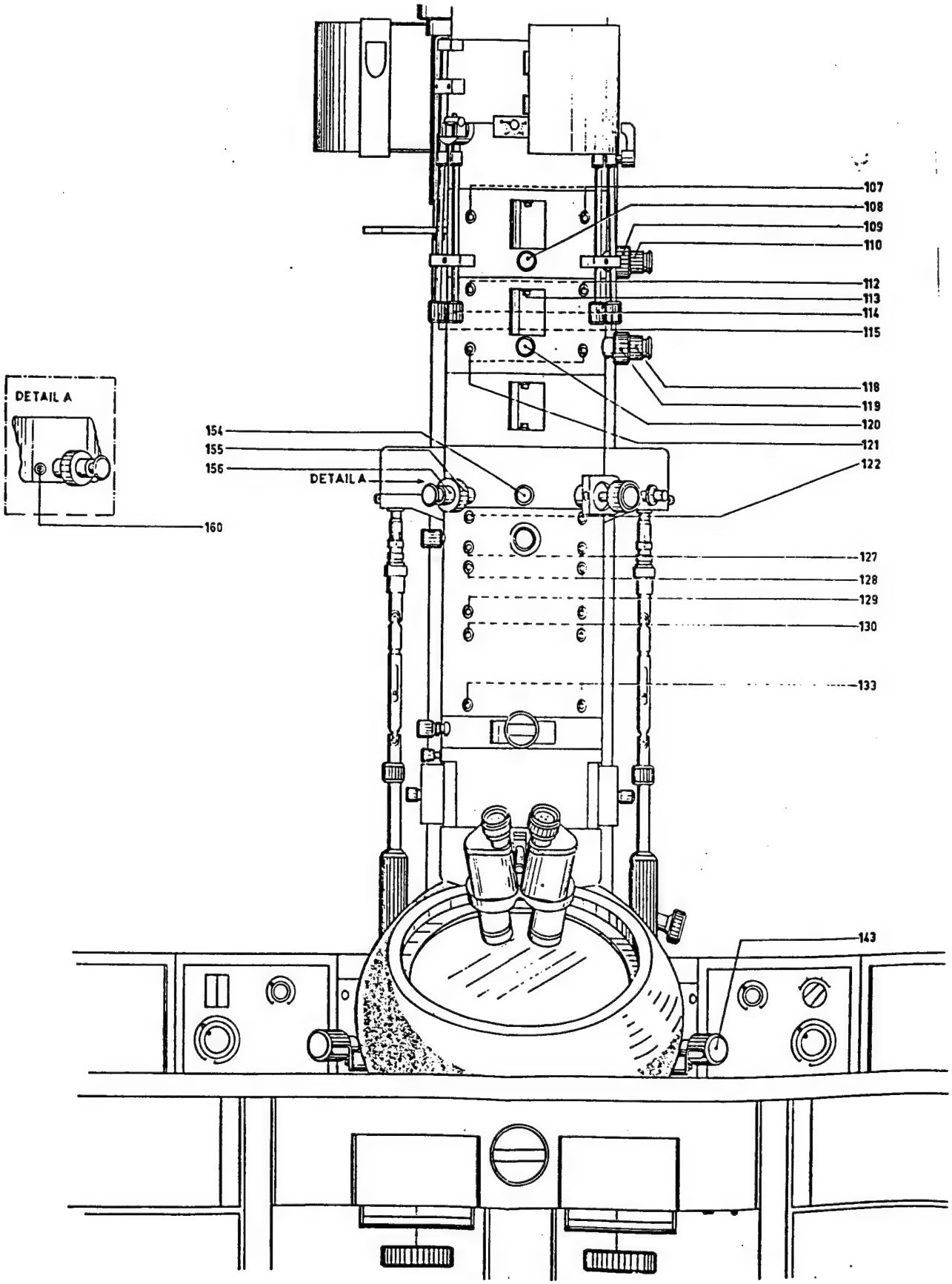


Figure 6 - Microscope Column Description

107	Adjustment screws for the upper polepiece of the first condenser lens
108 110	Fine adjustment controls of the first condenser lens aperture
109	Selector control for the aperture of the first condenser lens
112	Adjusting screws illumination system
113	Fixing screws for the illumination system
114	Lateral displacement of the electron gun
115	Tilting adjustment of the electron gun
118 120	Fine adjustment controls of the second condenser lens aperture
119	Selector control for the aperture of the second condenser lens
121	Adjustment screws for the lower polepiece of the second condenser lens
122	Adjustment screws for the lower polepiece of the objective lens
127	Adjustment screws for the diffraction lens
128	Adjustment screws for the lower polepiece of the diffraction lens
129	Adjustment screws for the upper polepiece of the intermediate lens
130	Adjustment screws for the intermediate lens
133	Adjustment screws for the projector lens
143	Specimen traverse controls (fine)
154 156	Fine adjustment controls of the objective lens aperture
155	Selector control for the aperture of the objective lens
160	Height adjustment control for the objective aperture

Introduction:

The microscope should be maintained under vacuum at all times. Exceptions include periods during which cleaning, maintenance, and loading and unloading of photographic films are taking place. Any internal parts that are exposed to vacuum should be handled with care. Use nylon, lint free gloves when internal parts are handled. Photo film boxes need not be handled with gloves, however, hands should be dry and free of obvious dust and dirt.

The microscope table should be kept free of extraneous materials. Only materials necessary for operation, alignment and data recording should be on the microscope table.

Care should be taken not to operate any control knobs in a random fashion.

The column and table of the microscope should be wiped clean with a damp cloth each week to maintain a clean environment and neat appearance.

Turning On The Microscope:

1. Turn on the water supply to the microscope.
2. Switch on the "Stand By" circuits (rear panel, No. 57).
3. Push the mains "On" button (right hand main panel, No. 1).
4. Press the "On" button which controls the vacuum system.
5. When the light "H.V." is extinguished, the vacuum in the column is adequate for operation and the high tension may be switched on.

Turning Off The Microscope

1. Turn the filament control all the way counter-clockwise.
2. Make sure the high tension is turned off (hand main panel, No. 10).
3. Push the mains "Off" button (right hand panel, No. 2).
4. Press the "Off" button which controls the vacuum system.
5. Switch off the "Stand By" circuits (rear panel, No. 57).
6. Set the timer for the water supply for 1 hour.
7. Turn the magnification function selector to "M" (right hand main panel, No. 24).
8. Reduce the magnification to position 1 (right hand main panel No. 23).
9. Overfocus the second condenser lens to position 17-20 (left hand main panel, No. 16).

Note: For overnight or weekend shut down, only perform steps 1-3.

Initial Control Settings (Only needed for inexperienced operator or microscope problems):

1. Switch off the first condenser lens (right hand auxiliary panel).
2. Switch on all other lenses.
3. Set the objective lens control to its middle condition.
4. Set the magnification function selector (No. 25) to position "M".
5. Overfocus the second condenser lens.
6. Turn the magnification control to position 1.
7. Ensure that the projector lens fine control (right hand auxiliary panel, No. 31) is in position "W".
8. Switch off the deflection system.
9. Remove the objective and diffraction apertures from the beam.
10. Withdraw the specimen into the airlock.
11. Set the emission control to position 6 or 7.
12. Set the specimen stage control micrometers (159) approximately to their middle positions.

Switching On and Adjusting The Illumination (Daily Alignment):

1. Switch on the high tension and wait until it has built up to its full value.
2. Increase filament control until saturation.
3. Set beam at crossover with condenser II controls.
4. Adjust gun lateral displacement knobs (114) to center beam.
5. Turn on deflection system and center beam with deflectors.
6. Desaturate filament, locate and center halo with gun tilt knobs (115).
7. Fine focus halo with condenser stigmators.

Switching Off The Illumination:

Illumination can be switched off by simply reducing the filament control to zero and pushing the high tension off button.

Placing and Removing Grid in Single Tilt Holder:

The grid is held in place by the modified spring loaded clamp forks, which must be raised before a specimen can be loaded.

1. Place sample holder horizontal in sample holder stand.
2. Raise the modified spring loaded clamping forks with the supplied tool and insert grid into holder sample side down, straight edge towards handle of sample holder.
3. Place beryllium cup on grid with flat side touching grid.
4. Lower modified spring loaded clamping forks onto beryllium cup with supplied tool.
5. Reverse sequence to remove grid.

Placing and Removing Grid in Double Tilt Holder:

The grid is held in place by a beryllium washer and nut. Proceed as follows:

1. Place sample holder horizontal in sample holder stand.
2. Place grid in sample holder sample side down, straight edge towards handle of sample holder.
3. Insert washer on top of grid (notice notches in washer).
4. Screw nut on top of washer and grid using supplied specialized tool.
5. Reverse sequence to remove grid.

Inserting The Sample Holder Into The Microscope:

1. Insert specimen grid holder into goniometer with pin on holder at 5 o'clock position.
2. Turn specimen grid holder anti-clockwise as far as it turns and guide it into goniometer. (Due to the scope being under vacuum the specimen grid holder will be sucked into the goniometer. Care should be given in order not to damage the specimen grid holder.)

Removing the Sample Holder from the Microscope:

1. Pull sample holder straight out of goniometer until it stops.
2. Turn sample holder clockwise until it stops.
3. Continue to pull sample holder out of goniometer.

Low Magnification:

1. Remove diffraction & objective apertures from the beam.
2. Set the magnification function selector switch (#24) to SC.
3. Set the magnification switch (#23) to some value below 11.
4. Fully overfocus the second condenser lens (coarse control 16 to position 26).

Working Magnification:

1. Set condenser 1 coarse control (#17) to position 3.
2. Set the magnification switch (#23) to position 14.
3. Set condenser II coarse control (#16) to 16.
4. Set the magnification function selector switch (#24) to "M".
5. Set objective lens (coarse control) to position 22.
6. Insert objective aperture.
7. Carefully focus the objective lens control (#25) for sharp images observed on the small viewing screen.

Obtaining an SAED Pattern:

In order to obtain accurate selection of the specimen area by the diffraction aperture, it is essential that the specimen should be accurately focussed in the plane of the aperture by the objective lens. This is done by first focussing the aperture on the screen with the diffraction lens and then focussing the image on the screen with the objective lens. The procedure is as follows:

To ensure that SAED patterns are viewed and recorded under the same parameters make sure initial control settings are set as follows:

In right hand auxiliary panel:

- Intermediate lens coarse control at position 4.
- Intermediate lens fine control at fully clockwise position.
- Diffraction lens coarse control at position 20 or 21.
- Diffraction lens fine control set so diffraction spot is at its smallest point.
- Projector lens coarse control at position 8.
- Projector lens fine control at fully anti-clockwise position.

Make sure the Z-axis is corrected on the goniometer stage.

1. Set the magnification function selector 24 to "SA".
2. Introduce and center the diffraction aperture using microscope column controls #123, #124 and #126.
3. Focus the diffraction aperture on the screen using the control "selected area" (#34) for the diffraction lens on the right hand auxiliary panel (use the small screen and binocular for this if required).
4. focus the specimen on the screen using the normal objective lens controls 25 (plus wobbler, binocular and small screen as required). The objective lens is now correctly adjusted for the relative positions of the specimen and diffraction aperture. It must not be altered or the accuracy of the selected area diffraction will suffer.
5. Switch the projector lens to W in the right hand auxiliary panel to view diffraction patter on small screen.
6. Switch the magnification function selector to "D+I".
7. If required, focus the diffraction pattern using the diffraction lens free range controls (#33).
8. Overfocus the second condenser lens to obtain a high quality pattern.
9. For amphibole SAED patterns it will be necessary to tilt the fiber in order to locate a zone axis. This is accomplished be engaging the tilt motor drive for the goniometer stage and turning it on via the motor control switch located on the microscope console.

10. To record a SAED pattern turn the projector lens fine control in right hand auxiliary panel fully anti-clockwise. Make sure condenser 2 lens is over focused. Place beam stop over central spot in diffraction pattern, remove small screen, lift up large screen, press the transport button (#40), set the exposure time to 32 seconds and press the exposure button (#39). Remove beam stop and expose for an additional .5 seconds. Lower large screen and press transport button (#40). Record photo number on data sheet and photo log book.

Obtaining EDS Spectra:

1. Make sure goniometer stage is eucentric (if not see section on Z-axis correction for goniometer stage.
2. Tilt sample 35 degrees towards detector.
3. Set condenser I to 26.
4. Set condenser II to 14.
5. Go to crossover.
6. Center beam with deflection system.
7. Remove objective aperture.
8. Push start on EDAX PV9900 operators console.
9. Collect spectrum for up to 90 seconds live time and store under sample batch number if necessary.
10. Profiles are compared to standard profiles if necessary.

Z-axis Correction For Goniometer Stage:

After changing specimens or even fields of view it is necessary to readjust the specimen height in order to ensure efficient use of the tilt feature:

1. Select a recognizable feature of the specimen and place it on the center of the screen using the stage controls.
2. Select a magnification, so when a large tilt of (30-40o) is applied to the specimen, the feature does not move off the screen.
3. Apply a tilt of 30-40o to the specimen and note the movement of the selected feature.
4. Adjust the height control until the feature returns to its original position.
5. Repeat steps 2-4 until the selected feature remains adequately stationary at the required magnification.

The Plate Camera:

This consists of two drawers into which the boxes for the exposed and unexposed plates fit, together with a mechanism built into the microscope for transporting an unexposed plate into position under the main screen and subsequently on into the box for exposed plates.

Taking a Micrograph (Non SAED Pattern):

Once an object has been selected for a photograph the exposure time can be determined by the exposure meter (#29 on right hand main panel) when selector switch (#30) is in position "EXP". The reading on the meter is altered by the operation of the exposure time selector (#38). When the meter reads half scale the correct exposure time is selected.

Loading The Film Box:

This must be carried out in a dark room under the appropriate safe light.

1. Fit plates into 16 plate holders of the appropriate size.
2. Remove the lid from the box for the unexposed plates.
3. Place the filled holders in a pile (plates uppermost) of the spring loaded platform of the box and press them down into the box taking care not to touch the surface of the plates with the fingers.
4. Holding the cassettes in place, fit the lid to the box and close it.
5. Put the box in the desiccator.

Fitting The Boxes To The Camera:

1. Close the valve and let air into the camera by pulling out and turning clockwise the plate camera airlock control (#145) as far as it goes about 1/2 turn. Pull the plate camera airlock control out as far as it goes (1-1 1/2 cm).
2. Close Box lids with plate box controls (#149) and release the two clamps (#148) for the plate drawers and pull the drawers open.
3. Fit both boxes to the drawers, taking care that the unexposed plate box goes into the right hand drawer and the exposed plate box into the left hand one.
4. In both cases, fit the pin #4 of the lid opening mechanism into the corresponding hole in the box so as to be able to open the box from outside the vacuum.
5. Close open drawers, and clamp the drawers in position. Open box lids with plate box controls (#149).
6. Pump down the camera chamber by turning the plate camera airlock control clockwise as far as it goes (45o) wait until the PVL light goes out then push plate camera airlock control in as far as it goes and turn it 1/2 turn until it stops.

Changing The Filament (EM300):

1. Reduce the filament control to zero and switch off the high tension.
2. Turn microscope handle #105 clockwise as far as it will go (about 3/4 turn, so it is vertically down).
3. Pull the handle out along its axis as far as it will go (about 2 cm).
4. Rotate the handle clockwise as far as it will go (about 45o).
5. Unscrew the high tension cable locking ring #102.
6. Using the cable lifter #106 lift the electron gun assembly out of the microscope column.
7. Remove the gun cap by pulling straight down and replace with a new one. Be sure to wear gloves during this procedure.
8. Using the cable lifter #106, lift the electron gun assembly back into the microscope column.
9. Screw on the high tension cable locking ring #102.
10. Turn handle #105 anti-clockwise as far as it will go.
11. When the PVL light is out, push handle in as far as it will go and turn it anti-clockwise as far as it will go.
12. When the HV light goes out the microscope is ready for use.

Replacing Filament In Gun Cap (Wehnelt Cylinder):

1. Disassemble Wehnelt cylinder and remove old filament.
2. Use Wenol to clean all parts of Wehnelt cylinder.
3. Use a Q-tip and Mr. Clean to remove as much Wenol from Wehnelt cylinder as possible.
4. Place all parts of Wehnelt cylinder in ultrasonic cleaner with Mr. Clean.
5. Replace Mr. Clean with distilled water and sonicate. Remove parts and blow dry with dust off (make sure lint free nylon gloves are worn for this entire process.)
6. Remove Wehnelt cylinder and center filament with allen screws. Screw cap back 9 divisions from having the filament flush with cap.
7. Wehnelt cylinder is now ready to be placed in electron gun.

Cold Trap:

The cold trap is filled with LN2 every morning and whenever need thereafter. This helps reduce burning of the specimens while under the beam (small spot size) for energy dispersive x-ray analysis.

B.5 Carbon Rod Sharpener Operating Procedure

1. Unscrew carbon rod holder nut.
2. Insert carbon rod so 2-1/2 inches sticks out of holder.
3. Screw down carbon rod holder nut.
4. Turn on unit by silver push button located in front of unit.
5. Put blade on guide.
6. Slowly bring blade into carbon rod.
7. Sharpen carbon rod to have about a 1 mm diameter point of approximately 5 mm long.
8. Turn unit off.
9. Unscrew carbon rod holder nut.
10. Remove rod by using push rod located on the side of the unit.

B.6 Carbon Evaporation Operating Procedure

1. Turn main power toggle on.
2. Be sure all switches on the lower half of the panel are set to "off" or "close", the filament selector is set to "left" and the thermocouple gauge is set to TC-1.
3. Vent bell jar using the switch marked "vent."
4. Lift the jar and place the samples on the rotating stage.
5. Place the sharpened rods in their holders and adjust the tension so that after the rods have been sputtered, they will almost touch.
6. Turn the rotating stage on briefly to ensure that the samples will not hit the bell jar when lowered.
7. Lower the bell jar and evacuate by switching from "vent" to "operate" mode. Wiggle the bell jar gently to seat it properly.
8. Wait until the vacuum is below 50 millitorr. If the carbon coater has not been used in the last few days, this may take more than 10 or 15 minutes. If pump down seems exceptionally long, vent the system and check the seal on the bell jar for debris.
9. Turn on the rotator and evaporate the carbon rods using the "flash power adjust" knob. This should be done slowly or in a series of 15 second bursts to keep the temperature in bell jar steady and to keep the pressure inside the bell jar low. As soon as the tip of the carbon has evaporated, turn the knob to off. Leaving the power on too long after the carbon tip has evaporated can seriously damage the carbon coater.
10. Turn off the rotator, vent the system, lift the bell jar and remove the samples.
11. To shut down the system, lower and evacuate the bell jar, pump down the system for 5 minutes and turn the power off.

B.7 Operation of SPI Plasma Prep II Plasma Asher

1. Turn on O_2 and bleed O_2 line.
2. Plug in vacuum pump.
3. Push in red AC button in lower left corner on front of asher, until light goes on.
4. Wait 10 minutes for system to warm up.
5. Place samples inside inner reaction chamber.
6. Flip up (on) vacuum switch.
7. Wait 2 minutes for inner reaction chamber to pump down to required pressure.
8. Flip up (on) meter switch.
9. Turn level knob fully clockwise.
10. Flip up (on) RF switch.
11. With tuning knob, tune for lowest reading on meter.
12. Ash samples for calibrated amount of time.
13. Flip down (off) RF switch.
14. Flip down (off) vacuum switch.
15. Wait 4 minutes for inner reaction chamber to come to atmospheric pressure.
16. Remove samples from inner reaction chamber.
17. Repeat if necessary.
18. Clean ad nd replace inner reaction chamber.
19. Unplug vacuum pump.
20. Push in red AC button in lower left corner on front of asher until light goes out.

B.8 Operation of Condensation Washer

1. Fill condenser chimney with fresh dry-rite (if desired).
2. Place several 3 mm glass beads in bottom of condenser.
3. Fill bottom of condenser 1/2 full with appropriate reagent for filters (acetone or chloroform).
4. Make sure stainless steel screen is properly installed on cold finger.
5. Make sure all hose connections are secure.
6. Turn on water to cold finger (use slow flow rate).
7. Make sure power supply is set on 120V.
8. Set output voltage to 35 for acetone or 45 for chloroform.
9. When some of the vapor has condensed at the level of the cold finger, place grids on stainless steel screen.
10. Make sure liquid-vapor line is at level of grids. Adjust water and power supply if change is needed.
11. Wash grids for appropriate times (see section on sample preparation).
12. After washing is complete allow grids to dry.
14. When grids are dry place grids in grid boxes and enter their sample number and grid box location in the grid log book.

B.9 Operation of the Film Desiccator

1. Place petri dish containing phosphorous pentoxide in bottom of desiccator (be very careful not to spill or touch this chemical - it causes severe burns).
2. Place all film and loaded sender box for microscope in desiccator and close door.
3. Close air inlet valve and open air outlet valve.
4. Turn on mechanical pump and evacuate desiccator for 30 minutes.
5. Close air outlet valve and turn off mechanical pump.
6. To open desiccator, unlatch door and open air inlet valve.

B.10 Ancillary Equipment

I. All instruments small and large used to prepare or analyze air samples must be on hand and in good operating conditions.

II. Surgical Knives:
Cutting air filters may be performed using a rocking motion of the curved surgical blade so that fibers are not disturbed or lost in preparation. Use of the zerostat gun is helpful to eliminate any static electricity. The surgical knives must be cleaned with filtered, deionized water or acetone between samples to control cross contamination.

III. Surgical Scissors:
Cutting air filters may be performed by using surgical scissors to cut the filter only after the filter has carefully been removed from the filtering cassette without disturbing any of the fibers. Use of the zerostat gun is helpful to eliminate any static electricity. The surgical scissors must be cleaned with filtered, deionized water or acetone between samples to control cross contamination.

IV. Tweezers:
Tweezers should have sharp tips that meet properly when operated and permit easy handling of air filters. The tweezers must be cleaned with filtered, deionized water or acetone between samples.

V. Glassware:
All glassware including prep dishes should be cleaned with Liquinox before each use and be free of any dust. Petri dishes should cleaned frequently to prevent contamination and to maintain proper acetone vapor pressure.

B.11 Calibration of the TEM

I. Introduction:
It is essential that the operating parameters of the electron microscope be known as accurately as possible. The two calibrations that are most significant with respect to measurement and identification are the magnification and the camera constant for selected area electron diffraction.

II. Magnification Calibration:
For magnification calibration on the screen and plate use a "waffle' type grid replica grating with 2160 lines per millimeter.

1. Insert the replica into the microscope and be sure that it is eucentric and in focus.
2. Perform the required on-screen measurements as indicated on the Magnification Calibration Form. The inscribed circles have diameters of 9mm and 90mm. Count the number of lines/spaces that are enclosed in each of the inscribed circles. Screen magnification is calculated using the formula below:

$$\frac{\text{circle diameter (mm) X 2160 lines/mm}}{\text{\# lines counted}}$$

3. The actual diameters (in um) of the circles can then be calculated as follows:
Large Circle:

$$\frac{90000}{\text{screen mag}}$$

Small Circle:

$$\frac{9000}{\text{screen mag}}$$

4. Photograph at low magnification (300X) and high magnification (15000-20000X). To determine the true magnification, perform the following calculation:

$$\frac{\text{Total Distance Measured (mm) X 2160 lines/mm}}{\text{number of lines counted}}$$

5. Record all the information (including the kV and TAP setting) and calculations on the sheet and on the control chart and archive the negative.

III. Camera Constant

Use the gold standard for this calibration and follow the instructions on the Camera Constant Determination form. (If the Aluminum standard is used, you must also obtain the proper d-values corresponding to the rings observed.)

1. Obtain an electron diffraction pattern (EDP) from a stable area of the gold standard. Be sure the sample is eucentric and in focus.
2. For on-screen calibration, insert the objective aperture and observe the number of rings that are eclipsed by the shadow of the aperture.
3. Photograph the EDP and measure the radii of the four smallest concentric rings several times and obtain an average value.
4. Using the known interplanar spacings for gold (listed on the sheet) calculate the camera constant as follows:

   Radius of ring 1 (mm) X 2.355 (A) = camera constant 1
   Radius of ring 2 (mm) X 2.039 (A) = camera constant 2
   Radius of ring 3 (mm) X 1.442 (A) = camera constant 3
   Radius of ring 4 (mm) X 1.230 (A) = camera constant 4

   The average of these four values is used as the camera constant.

5. Record all of the information (including kV and camera length) on the sheet and on the control chart and archive the negative.

B.12 Calibration of the EDAX

Insert the Cu-Al grid into the microscope. Turn on the EDAX. The calibration routine for the EDAX is located under ED SPEC on the main menu.

1. When asked which detector to use, choose detector 2.
2. For most applications, use the automatic calibration routine.
3. Peak Height = 6000.
4. Energy 1 = 1.486 (Aluminum).
5. Energy 2 = 8.040 (Copper).
6. Hit return, and the calibration procedure will run to completion.
7. If more than 6 iterations are required, the procedure must be repeated.
8. Record results of calibration in the EDAX calibration book.

B.13 Calibration of the Plasma Asher

Calibration of the low temperature oxygen plasma asher is carried out to determine the amount of time needed to ash 10% of the collapsed filter section.

1. Cut a filter section, place it on the slide and collapse it in acetone vapor.
2. Ash the collapsed filter section in the asher to completion. Divide this number by 10 to approximate the amount of time needed to ash 10% of the filter section.
3. Weigh a clean glass slide on the analytical balance.
4. Cut a filter section, place it on the slide and collapse it in acetone vapor.
5. Weigh the slide again and subtract the weight from step 4 to determine the weight of the collapsed filter section.
6. Ash the sample for the amount determined in step 2.
7. Weigh the slide again, subtract the weight of the empty slide, and calculate the percent weight loss. This should be 10%.
8. Record the time in the QC Log Book and post the time on the asher.

B.14 Preparation of Air Filters for TEM Analysis

1. Clean 2 glass slides (for a set of 5 inside air samples) and label them to correspond to the samples plus blanks.
2. Cut a wedge from each sample using the cork borer and attach each section to the slide using paper page reinforcers.
3. Place each slide in the collapsing chamber until the filters clear (about 4 minutes).
4. Place all of the slides in the plasma asher for the calibrated amount of time as posted on the asher.
5. Carbon coat the samples as described elsewhere in this document.
6. Cut 3 squares (2 x 2mm) from each filter section and place on copper grids (grids shiny side up).
7. Place grids on screens and place screens in the Jaffe Wick for at least 15 minutes.
8. Lift each grid from the screens and carefully place them on the quick wick for at least an hour or the condensation washer for at least 15 minutes.
9. Place the cassettes and grids in their respective storage areas.

Note: The above procedure is to be used for MCE filters. If PC filters are received, the same procedure is followed with the following exceptions:

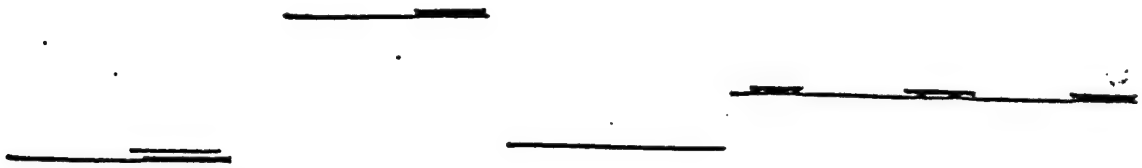
1. Filter sections are cut with a scalpel and attached to the slide with adhesive tape.
2. There are no collapsing or etching steps.
3. Use chloroform rather than acetone as the solvent.
4. Filter dissolution may require as long as two days. (It is not recommended to set up a chloroform condensation washer due to safety concerns.)

B.15 TEM Air Sample Analysis Procedure

1. Using the sheets generated from the sample log in, determine the number of grid square openings to be counted on each grid.
2. Insert the sample in the sample holder sample side down and oriented so that traverses at operating magnifications of about 18000X will run parallel to the grid square opening.
3. Turn on the microscope as described earlier in this document, and align the scope and calibrate the EDAX.
4. Assess the grid preparation at low mag and reject the grid if:
    a. Less than 50% of the grid is covered or less than 50% of the openings are not in tact.
    b. The replica is doubled over or folded across more than 50% of the grid. At least 20 grid square openings should be unobstructed.
    c. There is greater than 10% of undissolved filter material.
5. Choose a grid square opening with less than 5% holes and less than 25% particulate coverage and increase magnification to at least 18000X. Record which openings you analyze on the count sheet.
6. Counting should be performed in a series of parallel traverses. Each pass should overlap the previous one by at least 10%. After a structure is located and identified, the analyst returns to the original field of view and continues in the same traverse direction.
7. Counting rules:
    a. Any continuous grouping of particles containing an asbestos fiber with an aspect ratio of greater than 5:1 and length greater than or equal to 0.5um shall be recorded and counted as an asbestos structure.
    b. Each asbestos structure is further classified into the following structure types:
        Fiber: A structure having a minimum length greater than or equal to 0.5um and a minimum aspect ratio of 5:1 with "substantially parallel" sides.
        Bundle: A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter to the next fiber.
        Cluster: A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated, from the group. Groupings must have at least two intersections. (Intersection is defined as a non-parallel touching or crossing of fibers, with the projection having an aspect ratio >5:1).
        Matrix: A structure with a fiber or fibers with at least one end free and the other end embedded in or hidden by particulate. The exposed part of the fiber must meet the fiber definition.
8. Identification of the fibers as asbestos is accomplished through analysis of their crystal structure and their chemistry through electron diffraction and energy dispersive x-ray analysis. Descriptions of both types of analyses follow later in this document.

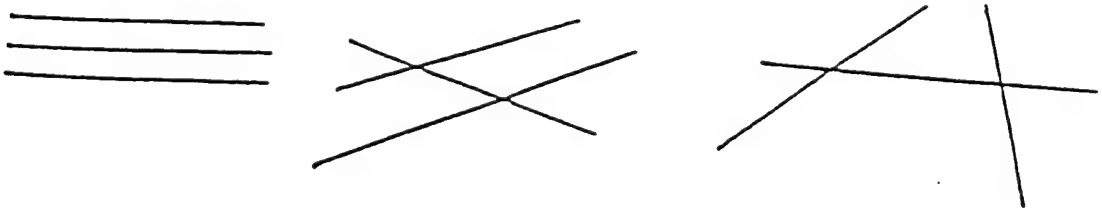
Figure 7 - AHERA Structure Sketches

Count as 1 fiber; 1 Structure; no intersections.



Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.

Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.

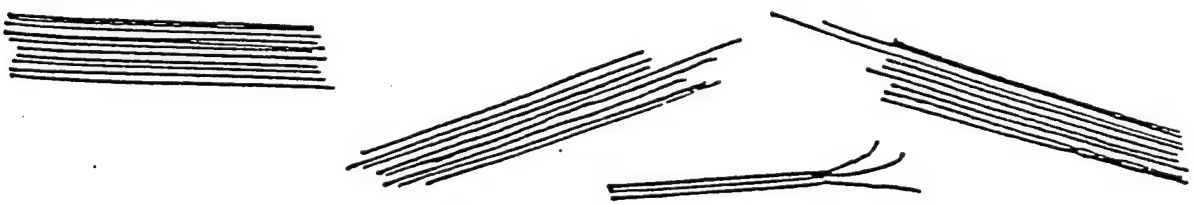
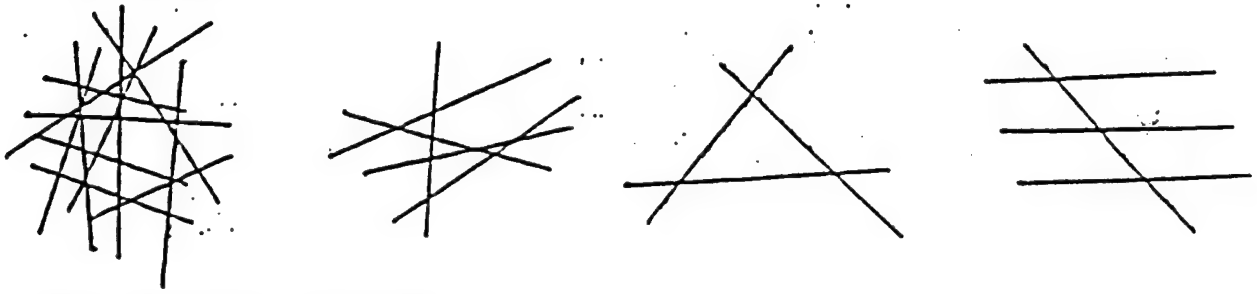
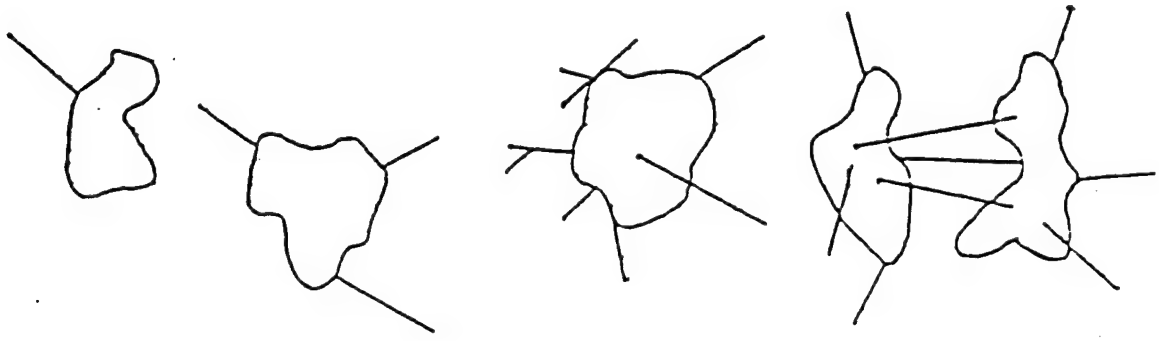


Figure 8 - AHERA Structure Sketches

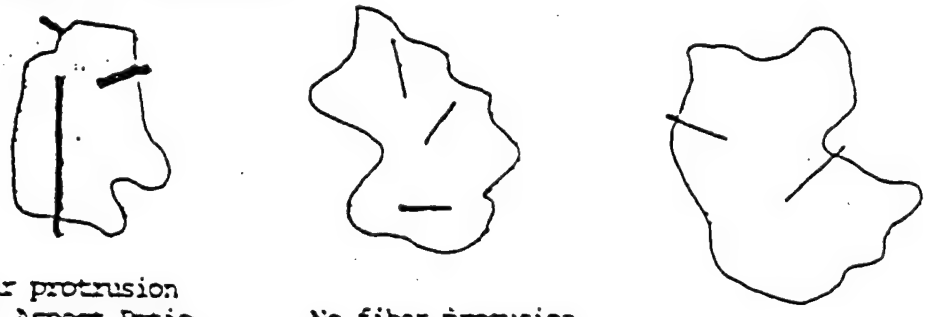
*Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTURES:



Fiber protrusion <5:1 Aspect Ratio

No fiber protrusion

Fiber protrusion <0.5 micrometer

—— <0.5 micrometer in length
— <5:1 Aspect Ratio

B.16 Selected Area Electron Diffraction (SAED)

An understanding of the mechanics behind measuring and interpreting SAED is required from each TEM analyst before identification of asbestos can be made. Countable structures are classified as asbestos by visual examination of their SAED pattern on the fluorescent screen using the calibrated markings on the screen or the diameter of the objective aperture for layer line reflection measurements and visual recognition of characteristic reflections. Both chrysotile and amphibole have layer line repeat distances of about 0.53nm. Amphiboles typically show closely spaced reflections with a high degree of symmetry depending on their orientation. Due to its cylindrical morphology, chrysotile typically shows streaking of the 110 and 130 reflections. As part of our quality control program, SAED patterns are routinely photographed and measured. The measurements of interest include row spacing, d-values of characteristic reflections and the angle between these reflections.

Indexing SAED Patterns of Chrysotile

1. Use the form provided and the following equation:

$$\text{d-value(A)} = \frac{\text{camera constant (mmA)}}{\text{distance mm}}$$

   a. Measure the distance between rows perpendicular to the row axes.
   b. Measure the distances from the central spot to the other reflections listed on the form and calculate the d-values using the equation above. If a reflection is difficult to measure near the central spot, use higher order reflections and divide the result accordingly.

2. Values acceptable for the primary reflections of chrysotile are:
   a. Row spacing: 5.17 - 5.45 A
   b. 110: 4.0 - 5.0 A
   c. 002: 7.06 - 7.54 A

Indexing SAED Patterns of Amphiboles

1. Row Spacing:
   a. The row spacing is the mean separation between horizontal rows and is measured perpendicular to row axes.
   b. Measure the distance (mm) between two adjacent rows. Also measure the distance between three rows and divide the number by two. Use an average of these two numbers in the calculation.
   c. The row spacing (A) is the camera constant divided by the distance measured in step b.
2. Reflection measurements within a horizontal row:
   a. From the SAED negative, determine the row with the most closely spaced reflections.
   b. Measure the distance (mm) between two adjacent reflections within the row. Also measure the distance between more reflections and divide by the appropriate number to obtain an

average distance between reflections within this row and several rows parallel to this row. Use the average in the calculation.

c. The d-spacing (A) of this refection equals the camera constant divided by the distance measured in step b.

3. Reflection measurements in other rows:
   a. Measure the distances and calculate the d-values of the reflections as described in 2a-c.
   b. Draw a line through the row being measured and the horizontal row in step 2 and using a protractor, measure the acute angle between the two rows.

4. Identification of the Zone Axis of the SAED pattern is accomplished through the use of these data compared to tabulated data from known amphiboles. These tables are kept in the laboratory and should not be removed from the premises.

3.17 Energy Dispersive X-Ray Analysis (EDXA)

To be classified as asbestos, countable structures must display EDX spectra closely matching spectra of known standards and or reference materials. By visually comparing the spectrum of the suspect asbestos to the spectrum of the appropriate standard, the analyst can classify the asbestos type on the basis of peak intensity ratios of the elements present. A semi-quantitative analysis may also be performed and compared to known values if deemed necessary.

Typical Asbestos Spectral Profiles:

	Na	Mg	Si	Ca	Fe
Chrysotile	0	7	10	0	0
Amosite	0	2	10	0	7
Crocidolite	1	1	10	0	6
Anthophyllite	0	3	10	0	1
Tremolite	0	4	10	3	<1
Actinolite	0	4	10	3	>1

Note: These are approximate relationships and can vary significantly. For example, the Mg:Si ratio of chrysotile can vary from 5:10 to 9:10.

B.18 Non Asbestos Minerals

Many fibers encountered during a typical TEM analysis may not be asbestos fibers and therefor not regulated. It is important to have the ability to distinguish these from asbestos easily during analysis. These minerals can be differentiated on the basis of their EDS and SAED data.

Gypsum - most easily distinguished by its chemistry (calcium and sulfur). It is often too thick to give rise to a readable SAED pattern.

Talc - fibrous and platy varieties. Since this mineral is similar in chemistry to chrysotile, a good EDXA is necessary. The Mg:Si ratio is lower in talc (.5 to .6) than in chrysotile (.7 to .9). The SAED for talc, however is significantly different than chrysotile. The pattern is typically hexagonal in the platy variety and streaked in the fibrous variety. Even though the pattern is streaked, a hexagonal array is usually apparent.

Vermiculite - contains aluminum.

Antigorite - polymorph of chrysotile. The fibrous variety of this mineral cannot be distinguished from chrysotile on the basis of chemistry. The SAED of antigorite will differ from chrysotile most obviously in the row spacing (.93nm for antigorite and .53nm for chrysotile).

Lizardite - another chrysotile polymorph. Does not generally occur as a fiber. SAED exhibits hexagonal geometry.

Sepiolite - not a cylindrical lattice. Therefor will not show streaking of SAED. Also has a much lower Mg:Si ratio than chrysotile.

Other minerals such as pyroxenes, halloysite, kaolinite and palygorskite/attapulgite are less common and can easily be distinguished from asbestos minerals by their SAED (pyroxenes have a b repeat equal to about half that of an amphibole) and their chemistry (halloysite, kaolinite and palygorskite/attapulgite all contain aluminum).

B.19 AHERA Recording Rules

1. Fill out all required information on the heading of the count sheets.
2. Identify the grids being analyzed as well as the grid openings in appropriate places on the count sheets.
3. Record NSD if no asbestos structures are detected in a particular grid opening.
4. List all asbestos structures in the order they are encountered.
5. Record the size information (>5um or <5um) and identification criteria in the appropriate columns.
6. Record at least one SAED and EDX spectrum of a typical asbestos structure per AHERA set. Any photos taken must be noted on the count sheet.

Chrysotile: Structures classified as chrysotile must be identified by SAED and EDXA and recorded on the count sheet. SAED or EDXA alone may be used as identification after a concentration of 70 str/sq mm has been exceeded for a particular sample. (Generally, this is after five structures have been counted.)

Amphibole: Structures classified as amphibole must be identified by SAED and EDXA and recorded on the count sheet. EDXA alone may be used as identification after a concentration of 70 str/sq mm has been exceeded for the sample.

Nonasbestos: SAED or EDXA alone may be used to identify a structure as nonasbestos. Record the presence of significant nonasbestos materials.

B.20 AHERA Stopping Rules

1. If more than 50 asbestos structures are counted in a particular grid opening, the analysis is terminated.
2. After counting 50 asbestos structures in a minimum of 4 grid openings, the final grid opening is completed and the analysis terminated.
3. After reaching the point where the average concentration for the 5 inside samples will exceed 70 str/sq mm, the analysis is terminated, the set fails initial clearance and the client should be notified. At this point the client must decide whether to re-clean and re-sample the area, or authorize the prep and analysis of the blanks and outside samples for a Z-Test.
4. For blanks, the analysis is always continued until 10 openings have been read.
5. In all other samples, the analysis is continued until a sensitivity of 0.005 str/cc is reached.

B.21 Calculations

1. To determine the number of grid square openings to be counted to reach an analytical sensitivity of 0.005 str/cc:

$$\frac{385 \text{ sq. mm}}{0.005 \text{ str/cc} \times \text{grid opening area (sq.mm)} \times \text{vol. sampled (cc)}}$$

2. To obtain the minimum detection limit (analytical sensitivity:

$$\frac{1 \text{ structure} \times 385 \text{ sq. mm}}{\text{\# grid openings counted} \times \text{grid opening area (sq.mm)} \times \text{vol sampled (cc)}}$$

3. To obtain the area analyzed:

$$\text{\# grid openings counted} \times \text{grid opening area (sq.mm)}$$

4. To obtain the concentration in structures per square millimeter:

$$\frac{\text{\# structures counted}}{\text{area analyzed}}$$

5. To obtain the concentration in structures per cubic centimeter:

$$\text{analytical sensitivity} \times \text{\# structures counted}$$

6. Mass calculations for EPA Level II analyses assume that both chrysotile and amphibole are cylindrical and that the width measurement is equal to the diameter of the cylinder. The densities of the minerals are 2.6 g/cc and 3.0 g/cc for chrysotile and amphibole respectively. Mass is calculated from the following equation:

$$\text{mass (ng)} = 3.14 \times (\text{length, um}) \times (\text{diameter, um})^2 \times (\text{density}) \times 10^{-6}$$

Bundle, cluster and matrix masses are calculated individually assuming a laminar structure with an average thickness equal to the fiber width. The individual masses are calculated using the following equation:

$$\text{mass (ng)} = (\text{length, um}) \times (\text{width, um})^2 \times (\text{density}) \times 10\text{-}6$$

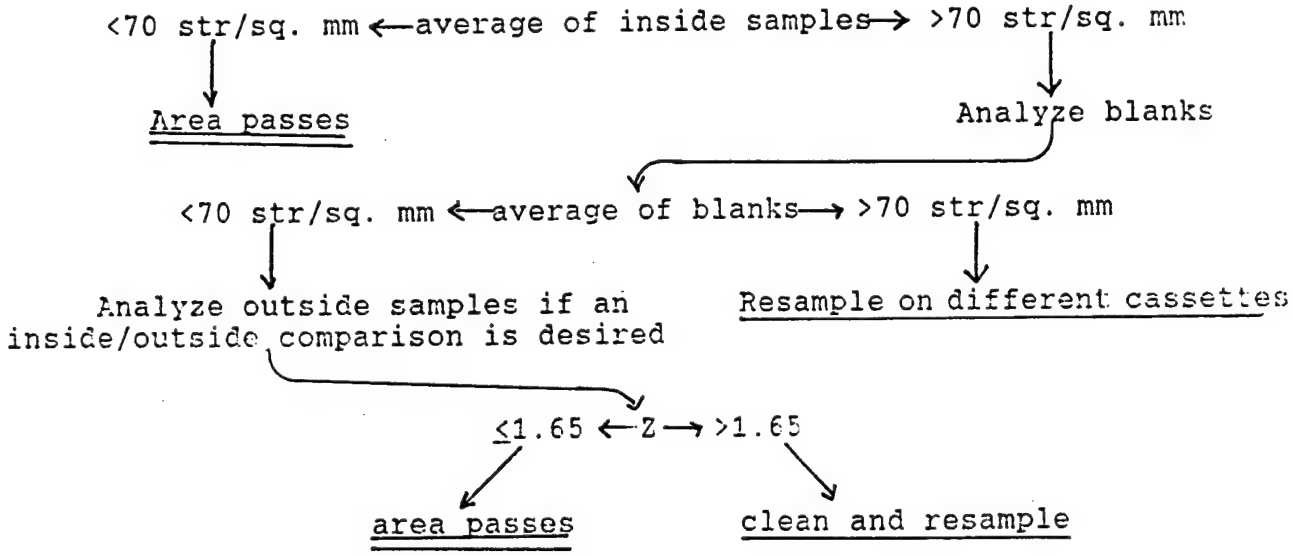
The total mass for the sample equals the sum of the masses each type of structure for each type of asbestos.

B.22 AHERA Clearance Criteria

The area passes if the average of the inside samples is less than or equal to 70 str/sq. mm. If this is not the case, then the blanks are analyzed. If their average is greater than 70 str/sq. mm, then there is a problem with contamination that may not be associated with airborne asbestos concentration in the abatement area. (If the lab blank is clean but the field blanks are not, the area should be sampled again using a different lot of prescreened cassettes. If the lab blank is dirty, the source of contamination is coming from the laboratory itself and must be traced using hood blanks, asher blanks, carbon evaporator blanks and wipes of tools and surfaces in the Prep area.) If the blanks' average is less than 70 str/sq. mm, the client must be contacted and asked if an inside/outside comparison is desired. If so, the outside samples area analyzed and a Z-Test is performed. If the result is less than or equal to 1.65, the area passes and the contractor is released. If not, the area must be recleaned and resampled.

Flow Chart for Determination of Completion of a Removal Response Action

Collect at least 5 samples inside and 5 outside the work area of at least 1199 liters for 25mm cassettes. Analyze according to AHERA regulations.



At any time, the client may decide to clean and resample rather than pay the additional expense of further analyses.

Z-TEST WORKSHEET

n_i = number of inside samples _____

n_o = number of outside samples _____

	Insides (str/cc)	ln	Outsides (str/cc)	ln
1	_____	_____	_____	_____
2	_____	_____	_____	_____
3	_____	_____	_____	_____
4	_____	_____	_____	_____
5	_____	_____	_____	_____
	Sum =	_____	Sum =	_____
	Y_i = sum/n_i =	_____	Y_o = sum/n_o =	_____

$Y_i - Y_o$ = _____

$\sqrt{1/n_i + 1/n_o}$ = _____ $0.8 \times \sqrt{(1/n_i + 1/n_o)}$ = _____

$$Z = \frac{Y_i - Y_o}{0.8 \times \sqrt{1/n_i + 1/n_o}} = \text{_____}$$

B.23 AHERA Reporting Protocol

The following information must be reported to the client for each sample analyzed:

1. Volume of air sampled.
2. Area of sample analyzed.
3. Number of asbestos structures detected.
4. Type(s) of asbestos detected.
5. Analytical sensitivity for the analysis.
6. Asbestos concentration (str/cc and str/sq. mm).
7. Copies of count sheets, spectra, etc.
8. Signature of laboratory representative indicating that the laboratory met the specifications of the method.

B.24 Sample, Grid and Data Storage

All air sample cassettes and grids shall be stored for a period not less than 1 year or returned to the client if requested. After that period, the cassettes may be disposed of.

Any original data generated from the analysis shall be stored with a copy of the report and filed indefinitely. Negatives from the samples are stored in protective sleeves and kept in a separate file with other negatives

B.25 Quality Assurance and Quality Control

Objectives

The analysis of asbestos air samples typically involves analyzing samples with nanogram quantities of asbestos. Consequently, contamination prevention is a critical factor in the preparation and analysis of these samples. Supplies, reagents, equipment and the analysts themselves are monitored on a regular basis to reduce the possibility of reporting erroneous results. Results of all QC measures are kept in the QC Data Book.

- Analyst accuracy is monitored to maintain a level better than 80% true positives (<20% false negatives) and less than 10% false positives.
- Laboratory blanks are prepared with each batch of samples and analyzed at least once per 25 samples analyzed.
- Duplicate and replicate analyses are performed at rate such that 10% of all samples analyzed are reanalyzed.
- Known materials (such as asbestos standards and SRMs) are analyzed on a regular basis.
- The lab participates in a round robin (interlaboratory) exchange program and performs proficiencies from several regulatory agencies on a regular basis.

Filter Lot Blanks

All TEM filters and sampling cassettes are ordered by and shipped to HASL. This is to ensure that appropriate lot blanks are drawn, analyzed and checked for contamination at a rate of at least 5%. Cassettes will then be distributed to the HGCL offices as they are needed. A filter lot blank average of less than 18 str/mm^2 with no single preparation exceeding 53 str/mm^2 is to be maintained. If this is not maintained and the contamination is indeed found to be in the filters and not from any other source, the samples and lot must be discarded. Sampling must then be done with a set of new sampling cassettes from a new lot of filters.

Field and Sealed Blanks

As described in the AHERA document, two field blanks (one inside and one outside the containment) and one sealed blank are required per clearance set. These blanks are taken at the time of sampling. They are analyzed after the inside samples and before the outside samples. The concentration of the field blank filters should be below 70 str/mm^2, and the concentration of the sealed blank should be below 53 str/mm^2.

Laboratory Blanks

All reagents and sample preparation equipment may be checked for contamination by the use of laboratory blanks which are prepared simultaneously with other samples. If a contamination problem is detected, individual reagents, tools and laboratory equipment can be checked separately to determine and correct the source of contamination. No sample results should be reported until a contamination problem is solved. The lab blanks' cumulative average concentration must be less than 18 str/mm^2 with a maximum of 53 str/mm^2 for any single preparation.

INTRALABORATORY QUALITY CONTROL

Analysis by Same Analyst: Four out of every 100 samples analyzed must be reanalyzed by the same analyst for a duplicate analysis. The same grid openings are analyzed each time. In addition, one sample out of every 100 analyzed must be reprepped and analyzed by the original analyst. Results of the analyses must fall within the guidelines specified in the appendix.

Analysis by Second Analyst: Four out of every 100 samples analyzed must be reanalyzed by a second analyst for a replicate analysis. The same grid openings are analyzed each time. In addition, one sample out of every 100 analyzed must be reprepped and analyzed by a second analyst. Results of the analyses must fall within the guidelines specified in the appendix.

Verified Analysis: This consists of each microscopist independently analyzing a grid square and comparing results. This requires that beam currents be low enough to sustain two consecutive analysis with electron diffraction patterns from the same fiber. Grid squares are counted in the same manner with identical grid orientations, starting point, and initial traverse directions. The size, number of structures, identification data, and location or drawing of these structures are recorded on the verified counting sheet. This data is compared to the multiple independent analyses and any questionable structures are to be reanalyzed and confirmed. The analysis will include at least one qualified microscopist with a proven average accuracy of greater than or equal to 5% false positives. The rest of the microscopists must attain an average accuracy equal to or greater than 80% of true positives, less than or equal to 20% false negatives, and an average of less than or equal to 10% false positives.

Analysis of Standards: Standards are analyzed by all microscopists on as needed basis or as required by the QC Frequency Chart. Any discrepancies detected may require that the analyst be further trained. Results of standard analyses are kept in the QC Data Book.

Analysis of NIST SRMs: SRM 2063a will be analyzed annually for each microscope. SRMs 1866 and 2076 will be analyzed quarterly by each analyst. Results are kept in the QC Data Book.

INTERLABORATORY QUALITY CONTROL

Interlaboratory testing consists of quarterly exchange of samples between three or more participating laboratories and comparison of results. This is commonly referred to as round robin analysis. In order to avoid the problems of preparing identical samples on filters for distribution among participants, prepared grids will be used. Each laboratory will prepare a total of two samples consisting of two grids each, to be analyzed according to AHERA rules with exceptions as noted. The number and location of grid openings to be analyzed should be indicated by the lab that sent the samples. Structures should be characterized according to AHERA rules and reported as structures per mm^2.

The preparation of filters is left to the discretion of each laboratory. It is not necessary for specimen grids to contain asbestos. The grids should be transported and stored in grid boxes with unambiguous labels. Grids should be measured and counted by each initiating laboratory and then passed on to the next laboratory in the sequence shown below. Samples should be considered as five-day turn around samples. The results of analyses should be transmitted to the next lab in the chain or to the host lab for statistical analysis. Comments about specimen preparation derived from examinations of material received are encouraged; there are none of us who cannot learn to improve our methods for preparing these materials. The selection of filter medium is at the discretion of the laboratory generating samples. Since NIST will probably require that both MCE and PC filters be prepared during any NVLAP on-site inspection, it would be prudent to vary filter type from time to time. Samples should be transmitted by Federal Express or equivalent to avoid unnecessary delays and to maintain a proper tracking record for each set of samples. The statistical analysis of the results together with comments on sample preparation will be presented to each laboratory in writing as soon as all information has been collected at the host lab.

QA/QC PASS/FAIL CRITERIA

There are two methods to determine whether or not a QC analysis passes. The method used depends upon the number of structures counted in the analysis.

For less than or equal to 20 structures counted in the QC analysis, use the following table to determine the upper and lower confidence limits:

Number of Structures Counted in QC Analysis	95% Confidence Limits	
	Lower	Upper
0	0.00	3.69
1	0.25	5.57
2	0.44	7.22
3	0.62	8.77
4	1.09	10.24
5	1.62	11.67
6	2.20	13.06
7	2.81	14.42
8	3.45	15.76
9	4.12	17.08
10	4.80	18.39
11	5.49	19.68
12	6.20	20.96
13	6.92	22.23
14	7.65	23.49
15	8.40	24.74
16	9.15	25.98
17	9.90	27.22
18	10.67	28.45
19	11.44	29.67
20	12.22	30.89

For more than 20 structures counted, use the following equation to calculate upper and lower limits:

(# structures) $\pm$ (1.96 X sq. root of the # of structures)

REPORTING OF RESULTS FOR AHERA SAMPLES

The following information must be reported to the client for each sample analyzed:

- Concentration of structures per square millimeter and structures per cubic centimeter.
- Analytical sensitivity for the analysis.
- Number of asbestos structures counted.
- Area analyzed.
- Volume of air sampled.
- Copies of count sheets.
- Signature of laboratory official indicating that the laboratory met the specifications of the method.
- For EPA Level II analyses, the asbestos mass concentration.
- Type of asbestos detected.

QA/QC FREQUENCY

QC Function	Frequency
System Check	Daily
Alignment Check	Daily
EDXA Calibration	Daily
Camera Constant	Weekly
Magnification Calibration	Monthly
Beam Dose Measurement	Monthly
Clean Air Monitoring	Quarterly
Asher Calibration	Quarterly
Water Level in Chiller	Quarterly
Spot Size Verification	Quarterly
Detector Resolution	Quarterly
SRM 1866	Quarterly
SRM 2063	Annually
K-Factors	Annually
Same Analyst Reanalysis	4 in 100 Samples
New Analyst Reanalysis	4 in 100 Samples
Repeat Prep and Analysis by Same Analyst	1 in 100 Samples
Repeat Prep and Analysis by New Analyst	1 in 100 Samples
Screen Cassette Filters	1 in 50 Received
Lab Blank Prep	1 per AHERA Set
Lab Blank Analysis	1 per 5 AHERA Sets
Record and Verify EDP on Negative	1 in 5 Pos. Samples
Review Field Data Sheets	Each Sample
Review Chain of Custody	Each Sample
Hand Recalculation of Data	1 in 100 Samples
Review Report	Each Report Generated
Supplies and Reagents Screening	As Needed
Evaporator Bell Jar Cleaning	As Needed
Grid Square Opening Area	As Needed

PART C

PHASE CONTRAST MICROSCOPY

C.1 PCM Laboratory Operation - General

I. FUNCTION:

The use of phase contrast microscopy (PCM) for the analysis of airborne asbestos is well documented in the NIOSH Method 7400. The method will not distinguish between asbestos and non-asbestos fibers but it will permit counting of fibers greater than 5 micrometers in length and greater than .25 micrometers in diameter.

II. CONTROL

1. To assure accurate and precise results the analytical sequence, equipment, reagents, and analysis must be quality controlled.

2. Materials that might produce spurious data should not be introduced into the laboratory.

3. Controls of procedures should be limited to the Laboratory director and/or his designee and the rules of operation and maintenance should be strictly adhered to.

4. Individual operators should be thoroughly trained and be under the supervision of an experienced microscopist.

C.2 Quality Assurance Objectives

The objective of this document is to assure that all data are precise, accurate, complete, representative and comparable in order to satisfy the NIOSH 7400 Method.

In order to meet this objective, procedures have to be implemented that strictly control collection of samples, sample handling and shipment, sample receiving, sample preparation, sample analysis, data reduction, reporting of results, record keeping, and performance checking.

1. HASL's precision for each analyst for replicate fiber counts should be documented by:

   A. Maintaining as part of HASL's Quality Assurance Program a set of reference slides to be used on a daily basis. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and PAT samples. The Quality Control Coordinator should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.

   B. From blind repeat counts on reference slides, estimate HASL's intra- and inter-analyst relative standard deviation (Sr). Obtain separate values of relative standard deviation for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, 21 to 50 fibers in 100 graticule fields, 51 to 100 fibers in 100 graticule fields, and 100 fibers in less than 100 graticule fields. Maintain control charts for each of these data files. Note: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision.

2. Prepare and count field blanks along with the field samples. Report counts on each field blank. The identity of field blank filters should be unknown to the counter until all counts have been completed. If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

3. Perform blind recounts by the same counter on 10% of filters counted (slides should be counted several hours later or the following day). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: discard the sample if the difference between the two fiber counts exceeds 2.77 x

(F)(Sr) where F = average of the two fiber counts and Sr = intra-analyst relative standard deviation from Step 1 B. Note: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

4. Enroll each new counter in a training course which compares performance of counters on a variety of samples using this procedure. Note: HASL is participating in the NIOSH Proficiency Analytical Testing Program (PAT) and is routinely exchanging field samples with other laboratories to compare performance of counters.

C.3 Staff Assignments

1. When the samples arrive at HASL, the log-in clerk checks the samples and data sheet/Chain-Of-Custody for completeness and requirements before initiating the analysis.

2. The QA/QC coordinator checks all laboratory reagents and supplies to meet specifications. (i.e, all reagents must be ACS reagents or better.

3. The analyst checks the cleanliness of the facility on a periodic basis by prepping and analyzing a laboratory blank along with a batch of air samples. (1%)

4. All PCM preparations are done in a clean environment away from the bulk analysis laboratory.

5. Daily, the analyst must perform a system check on the Phase Contrast Microscope before he/she analyzes actual samples.

6. The analyst must calibrate the Walton Beckett Graticule with a stage micrometer on a monthly basis and must determine the phase-shift limit of the microscope on a daily basis. These data are recorded in the microscope log-book.

7. The QA/QC coordinator will ensure that the analyst is qualified to perform analysis by evaluating on a continuous basis the following parameters:

   A. Replicate Counts
   B. Duplicate Counts
   C. Known Samples Counts
   D. PAT Samples Counts
   E. Interlab Round Robin Counts

8. The QA/QC coordinator will validate all data entries.

9. The QA/QC coordinator will recalculate 1% (one percent) of all computations and automatic data reduction steps as specified in the summation table.

   As soon as the results have been checked by the PCM Manager, the analyst will draft a report using the format as shown below. The analyst should check for accuracy of typing of results and text. Once the analyst is satisfied the report is error-free, he/she signs the report and forwards the report to the PCM Manager for review. After the PCM Manager has reviewed the report, it is forwarded to the Laboratory Director for his approval and signature. The report is then given to the log-in clerk for reproduction, filing, and mailing.

C.4 Sampling Procedure

1. Calibrate each sampling pump with a representative sampler in line.

2. For personal sampling, fasten sampler to the worker's lapel near the workers's mouth. Remove top cover from cowl extension (open face) and orient face down. Wrap joint between cowl and monitor body with electrical tape to prevent air leaks. Note: If possible, ground the cassette to remove any surface charge, using a wire held in contact (e.g., with a hose clamp) with the conductive cowl and a non-electrical metal fixture, or a cold-water pipe.

3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Remove top covers from the field blank cassettes and store top covers and cassettes in a clean area (bag or box) with the top covers from the sampling cassettes during the sampling period. Replace the top covers in the cassettes when sampling is completed.

4. Sample at 0.5 LPM or greater. Adjust sampling flow rate, Q (LPM), T (MIN),, to produce a fiber density, E, of 100 to 1300 fibers/mm^2 (3.85x104 to 5x105 fibers per 25-mm filter with effective collection area AC = 385 mm^2 for optimum accuracy. These variables are related to the action level (one-half the current standard), L (fibers/ML), of the fibrous aerosol being sampled by:

$$T = \frac{(AC)\ (E)}{(Q)\ (L)\ (103)}$$

Note: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 LPM for 8 hours is appropriate in non-dusty atmospheres containing ca. 0.1 fiber/ML. Dusty atmospheres require smaller sample volumes (<400 L) to obtain countable samples.

In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 LPM) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/ML, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If >50%

of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

5. At the end of sampling, replace top cover and small end caps.

6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

7. Record the following information onto the Data Sheet/Chain of Custody Form.

   - Start Time
   - Start Flow Rate
   - End Time
   - End Flow Rate
   - Sample ID Number
   - Type of Samples (Area/Personal)
   - Location of Sample
   - Comments (things that might affect the sample)
   - Average Flow Rate
   - Total Time of Sample
   - Client's Name
   - Job Number
   - Batch Number
   - Date Sample Taken
   - Name of Person Taking The Sample
   - Print Name and Sign Chain of Custody
   - Write the Date and Time Chain of Custody

C.5 Alignment And Calibration Procedures

1. Turn on the power and adjust potentiometer so that light is comfortable for your eyes.

2. Place a slide on the mechanical stage and place 10x objective into the optical system. Focus an object on the slide.

3. Close the field iris until it's about the size of the Walton-Beckett Graticule. Adjust the height of the condenser to its optimum location by making the edges of the polygon as sharp as possible. Ascertain that the light is going through the center of the optical system by centering the polygon with the two condenser centering screws.

4. Place the 40x objective into the optical system. Adjust light for personal comfort and focus on an object on the slide. Check the polygon of the field iris for centration and correct condenser height. If needed, fine tune the condenser height adjustment and the two condenser centering screws.

5. Open the field iris until the polygon just disappears.

6. Open the condenser iris completely - turn the numerical aperture to 1.25.

7. Remove one of the eyepieces and install the centering telescope in its place. While holding the body of the centering telescope stationary and rotating the viewing end, unscrew the viewing end from its main body until the perceived ring is as sharp as possible.

8. Grasp the phase annulus (which is beneath and within the condenser) with the thumbs and index fingers of both hands and make the rings concentric.

9. Remove the centering telescope and replace with the eyepiece. Set the interpupillary distance for your eyes by grasping the binocular head with both hands and adjusting the distance between the two eyepieces until the field of view appears as one field and not overlapping fields.

10. To adjust for differences in each eye the diopter adjustment is made. With the left eye closed, the fine focus is used to focus on an easily discernible particle. With the right eye closed, the left eye views the same particle and makes it as sharp as possible by adjusting the diopter adjustment.

11. The phase-shift detection limit of the microscope is

checked by using the HSE/NPL phase contrast slide and the procedures as set forth in the NIOSH 7400 Method.

12. Alignments and phase-shift detection limit of the microscope are done daily and more frequently if there are any questions of its adjustments.

13. The Walton-Beckett Graticule is calibrated monthly with a stage micrometer.

14. Documentation of the microscope calibration and alignments adjustments are registered by the analyst initializing the microscope calibration logbook.

C.6 Equipment Maintenance Procedures

1. Whenever visible dust is evident on the optical system, it should be cleaned by a gentle spray of clean freon gas and/or with a lint-free tissue. If solvent is indicated, then ethanol applied to a lint-free tissue should be used and care should be exercised not to drip any excess solvent onto any adhesive matrix that may over time loosen part(s) of the optical system.

2. The non-optical parts of the system should be maintained in a state of cleanliness by wiping with a wet lint-free cloth.

3. When the halogen bulb needs to be replaced, the new bulb should only be handled with a tissue paper - minimizing any transmission of body oil onto the bulb's glass envelope. If oil or other contamination does touch the bulb, it will shorten bulb life.

PREVENTATIVE MAINTENANCE CHECKLIST

Item	Frequency	Failure Action
PCM Microscope		
Oil Movable Parts	As Needed	
Clean Lens	As Needed	
Light	As Needed	Change Bulb
Wet-Wipe Other Parts	As Needed	
Acetone Vapor Generator		
Septum - Change	As Needed	Change Septum
Acetone Path	Poor Deposition	Remove Septum and Flush with Compressed Freon
Dual Counter		
Oil Movable Parts	As Needed	
Syringes	As Needed	Obtain New Syringe and Dispose of Old One as Prescribed by Local Ordinances
Scalpels	As Needed	Change Blades

ANCILLARY EQUIPMENT AND PROCEDURES

Acetone Vapor Generator

Plug instrument into an electrical outlet and switch on. While the heating block is getting hot, the filter membrane is positioned directly beneath the acetone outlet. With the syringe, the analyst draws approximately 250 ul of acetone and pierces the generator's intake port septum. When the light comes on, the analyst at first, slowly injects the acetone into generator while watching below at the exhaust port to make certain that the electrical charges between the exhaust port and the membrane is not so great that the membrane is pulled to the port before it is wetted to the slide by the initial gentle acetone mist. Once the initial mist has tacked the membrane onto the slide, the remaining acetone can be injected into the generator.

Syringes

Separate syringes are used to meter out small volumes of acetone and triacetin in the preparation of slides for phase contrast microscopy.

Scalpels & Tweezers

Scalpels and tweezers are tools which the analyst uses to manipulate and cut the mixed cellulose ester membranes in the preparation of microscope slides.

Microscope Slides, Cover Glass, and Lens Tissues

Although HASL uses precleaned 1mm thick 75 x 25 mm microscope slides and No. 1 1/2 22mm x 22mm cover glasses, the analyst does use lens tissue paper to wipe off all microscope glassware and manipulation tools between samples to minimize cross-contamination. Additionally, wiped slides are placed on top of a clean lens tissue to prevent its intimate contact with any potential dirty surfaces.

Dual Counter

The analyst uses a dual counter to keep track of both the fields and the fibers counted.

C.7 Sample Preparation and Analysis Procedures

Sample Preparation:

Note: The object of this procedure is to produce samples with a smooth (non-grainy) background in a medium with refractive index <1.46. This method collapses the filter for easier focusing and produces permanent mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" technique may be used outside the laboratory.

1. Ensure that the glass slides and cover slips are free of dust and fibers. They should be wiped clean with lint-free tissue.

2. The scalpel and forceps should be cleaned with lint-free tissue after each sample to prevent cross-contamination of samples.

3. With a permanent marker, and with the frosted end on the right, write lab ID Number at the top for the left sample and at the bottom for the right sample. Great care must be exercised not to mix up the samples. A good practice is to keep the cassettes orientated left - right and to use the same positions for the membranes on the actual slide.

4. Mount a wedge cut from the left sample filter onto a clean glass slide.

   A. Cut wedges of ca. 25% of the filter area with a curved-blade steel surgical knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide. Note: static electricity will usually keep the wedge on the slide.

   B. Place slide with wedge onto the acetone vapor generator slide holder and push the slide holder into the acetone vapor generator with the wedge centered directly below the acetone ejection port. Puncture the septum of the acetone vapor generator with the tip of a syringe containing ca. 250 ul acetone. Inject the acetone onto the vaporization chamber with a slow, steady pressure on the plunger button while holding the syringe firmly in place. After waiting 3 to 5 seconds for the wedge to clear, remove the syringe and slide from the acetone vapor generator. Caution: Although the volume of acetone used is small, safety precautions are necessary. Work in a well-ventilated area. Take precautions not to ignite the acetone. Continuous, frequent use of this device in an unventilated space may produce explosive acetone vapor concentrations.

D. Using a 5 ul syringe, immediately place 3.0 to 3.5 ul triacetin on the wedge(s). Gently lower a clean cover slip onto each wedge at a slight angel to reduce bubble formation. Note: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.

E. In order to make a mount permanent, glue the edges of the cover slip to the slide using lacquer or nail polish. Counting may proceed immediately after clearing and mounting are completed.
Note: If clearing is slow, warm the slide on a hot plate set on a low setting for up to 15 min to speed clearing. Heat carefully to prevent gas bubble formation.

Sample Analysis:

1. Center the slide on the stage of the calibrated and aligned microscope under the objective lens. Focus the microscope on the plane of the membrane.

2. Select "A" rule unless requested otherwise.

   A. Rules

      1. Count only fibers longer than 5 um. Measure length of curved fibers along the curve.
      2. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
      3. For fibers which cross the boundary of the graticule field:
         A. Count any fiber longer than 5 um which lies entirely within the graticule area.
         B. Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of Rules A.1 and A.2.
         C. Do not count any fibers which cross the graticule boundary more than once.
         D. Reject and do not count all other fibers.
      4. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
      5. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.

   B. Rules
      1. Count only ends of fibers. Each fiber must be longer than 5 um and less than 3 um diameter.
      2. Count only ends of fibers with an aspect

(length-to-width) ratio equal to or greater than 5:1.

3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets the Rules B.1 and B.2.
4. Count visibly free ends which meet Rules B.1 and B.2 when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 um in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets Rules B.1 and B.2.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 graticule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

3. Start counting from the tip of the membrane and progress along a radial line to the outer edge. Shift up or down on the membrane, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum each analysis covers one radial line from the membrane center to the outer edge of the membrane. When an agglomerate covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not count the rejected graticule fields in the total number counted.

Note 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.

Note 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestos, there is presently no acceptable method for ensuring uniformity of judgment between laboratories. It is therefore the policy of this laboratory using the method to report total fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample.

C.8 Statistical Information

Definitions:

DATA - A collection of information representing fact.

MEAN (X) - The arithmetic average. The sum of the data divided by the number of data.

MEDIAN - When the data are arranged in numerical order, there will be a number which will be in the center of the data.

NORMAL DISTRIBUTION - When the mean, mode, and the median are the same (or nearly so), the data assumes symmetrical, bell-shaped distribution which is called a "normal" distribution.

ACCURACY - A measurement which is said to be accurate implies that the measurement is correct.

PRECISION - Precision implies reproducibility.

SAMPLE - A sample is data obtained from a portion of the group defined as the population. The sample should be of sufficient numbers of individuals so as to represent the population.

CONFIDENCE LIMITS - Is a mathematically-determined interval, defined, by upper and/or lower limits, for which one is sure (with some degree of confidence - usually 95%) that the true value is greater than, less than, in or between.

STANDARD DEVIATION(S) - The standard deviation is a number which indicates the scatter of the data from the mean. It can be used in a variety of equations to predict, with different degrees of confidence, various limits. It is calculated as follows:

1. Determine the mean and subtract the mean from each piece of data. This is the deviation of the datum from the mean.
2. Square the deviation. This rids the calculation of + and - deviations. If one number deviates +2 and another deviates -2, the average deviation would be 0 which implies there is no deviation.
3. Sum the deviations squared and divide the sum of the deviations squared by the number of data less 1.
4. Take the square root.

COEFFICIENT OF VARIATION (CV) OR THE RELATIVE STANDARD DEVIATION

CV = STANDARD DEVIATION / MEAN

Statisticians prefer to leave the CV as a decimal value. The CV may be multiplied by 100 to convert it to a percentage value. The CV expressed as percent helps visualize the amount of variability. When the CV is multiplied by the mean, the result is the standard deviation.

STANDARD DEVIATION = CV X MEAN

C.9 Quality Control

The purposes of determining, using, and enforcing control limits is to increase precision.

When one is dealing with a specific set of data, it is easy to calculate and even to intuitively visualize specific statistics about the sample. One can look at the data and determine 95% of the data falls within a certain range. But one purpose of statistics is to collect a "sample" group of data, measure statistical items of interest, and then based upon the sample, predict the future performance of similar data. In quality assurance, one wishes to control a procedure (or measure the degree of control) to within two kinds of limits:

CONTROL LIMITS - UPPER & LOWER 95% CONFIDENCE LIMITS

95% of the time, one might want to limit counting acceptability to certain "control" limits. Measurements less than or greater than the control limits would be deemed unacceptable or inappropriate. Keep in mind that the control limits are at 95% confidence. There is always a small probability (5%) that some one may be high or low (meaning outside the control limits on occasion). Someone consistently being outside the control limits has a possible problem which should be identified and corrected. Control limits predict, within 95% probability, a range between which the data should fall.

CONTROL LIMITS (2 SIDED)

UPPER CONTROL LIMIT = MEAN + (1.965 X S)

LOWER CONTROL LIMIT = MEAN - (1.965 X S)

WARNING LIMITS - UPPER & LOWER 68% CONFIDENCE LIMITS

Warning limits are criteria for determining when the data are beginning to be too high or low. Data within the warning limits are considered to be good. Warning limits calculate a range of numbers in which 68% of the data should fall (based upon a previous statistical evaluation of the method).

WARNING LIMITS (2 SIDED)

UPPER WARNING LIMIT = MEAN + (1.000 X S)

LOWER WARNING LIMIT = MEAN - (1.000 X S)

HASL'S RELATIVE STANDARD DEVIATION (CV)

It is important to determine (both intra and interlaboratory) HASL's values of relative standard deviation (CV). This will be done over a period of time by having each analyst count reference slides. The reference slides are initially composed

of 5 slides: one slide is between 5 to 20 fibers per 100 fields; one slide is between 21 to 50 fibers per 100 fields; one slide is between 51 to 100 fibers per 100 fields; and one slide is 100 fibers in less than 100 fields. Several slides are counted on a daily basis until approximately 30 data per slide are obtained. On the x-axis is the number of fibers counted per 100 fields while on the Y-axis is the relative standard deviation (CV). The CV is calculated from the equation:

$$CV = S / MEAN$$

The "CV" versus "fibers counted" curve is then best fitted to the total data. Until the HASL does establish its own relative standard deviation (CV) for fiber counting by Phase Contrast Microscopy, HASL will use the CV as given in the following Figure (e.g., total fiber count = 10, CV = 0.40; and total fiber count = 100, CV = 0.114). The equation representing this curve is as follows:

$$CV = [\ (CV1)^2 + (.05)^2\ ]^{.5}$$

WHERE CV1 = ANTILOG10 [.0595 - .3241LOG FB - .01585LOG2 FB] - .12

where FB = fiber count per 100 fields.

The relative standard deviation plays a very important role in quality control and data reporting.
It is useful to estimate the 90% confidence interval on the mean count from a single fiber count. These "CV" vs. "Total Fibers Counted" curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results. (For example, if a sample yields a count of 24 fibers, Figure 1 of Method 7400 indicates that the mean interlaboratory count will fall within the range of 227% above and 52% below that value 90% of the time. These percentages apply directly to the air concentrations as well. If for instance, this sample (24 fibers counted) represented a 500 liter volume, then the measured concentration is .02 f/cc (assuming 100 fields counted, 25-mm filter, .00785 MM^2 counting field area.) If this sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.05 f/cc. These limits should be reported in any interlaboratory QC analysis.)

The relative standard deviation is also used to check the validity of replicate counts by the same analyst. This check of paired counts is done by using the following equation:

$$F2 - F1 > 2.77\ (AF)\ (CV)$$

Where F2 = Replicate Count; F1 = Initial Count; AF = Average of F2 + F1

If the difference of the paired counts exceed the product of

the paired count average, the relative standard deviation, and 2.77 then the paired counts are rejected and the sample is recounted.

The relative standard deviation is also used to track HASL's quality control of the Phase Contrast Microscopy procedure. On a weekly basis HASL's CV is determined and plotted in a continuously updated CV versus time control chart. The chart is composed of 5 parameters:

1. The Mean
2. Upper Warning Limit
3. Lower Warning Limit
4. Upper Control Limit
5. Lower Control Limit

These 5 parameters can be equated by the following:

MEAN (X) = STANDARD DEVIATION / CV = S / CV = X

UPPER WARNING LIMIT = X + (1) (S) = X + (X) (CV)

LOWER WARNING LIMIT = X - (1) (S) = X - (X) (CV)

UPPER CONTROL LIMIT = X + (1.965) (S) = X + (1.965) (X) (CV)

LOWER CONTROL LIMIT = X - (1.965) (S) = X - (1.965) (X) (CV)

SUMMARY OF PCM LABORATORY DATA QUALITY OBJECTIVES

OPERATION	QC CHECK	FREQUENCY	CONFORMANCE EXPECTATION
Sample Receiving	Review of Receiving Report	Each Sample	95% complete
Sample Custody	Review of Chain-Of-Custody Record	Each Sample	95% complete
Sample Preparation	Supplies & Reagents	On Receipt	Meet Specs or reject
Laboratory Blank		1 per 100	Meet Specs (<0.1 fibers per Walton Beckett) or reanalyze series
Sample Analysis	System Check	Each Day	100%
	Walton Beckett Graticule Calibration	1 Per Mo	95%
	Phase-Shift Detection Limit	Each Day	95%
Performance Check	Laboratory Blank (Cleanliness)	1 Per Mo	
	Replicate Count (Precision)	1 Per 10	95%
	Duplicate (Reproducibility)	1 Per 10	80%
	Known Samples (Working Std)	Training & Comparison	100%
	Pat Analysis	4x Per Yr	100%
	Interlab Round Robin	2x Per Yr	100%
	Data Entry Review (Data Validation & Measure of Completeness)	Each Sample	95%
Calculations and Data Reduction	Hand calculations of Automated Data Reduction Procedure or independent recalculation of hand-calculated data	1 Per 100	90%

C.10 Data Reduction and Analysis

I. Limit of Detection (Estimated) = 7 fibers per mm^2

II. Limit of Quantification = 13 fibers per mm^2
= 10 fibers per 100 fields

III. Range = 100-1300 fibers per mm^2

IV. Concentration =

$$\frac{\text{Average Count} \times \text{Sampling Area}}{\text{Field Area} \times \text{Flow Rate} \times \text{Time} \times \text{Conversion Factor}}$$

Where:

Avg Count = Fibers Per Field

Sampling Area = 385 mm^2 (For 25mm diameter Cassette)

Field Area = 0.00785 mm^2 Per Field

Flow Rate = Liters Per Minute

Time = Minutes

$$\text{Conversion Factor} = \frac{1000cc}{L}$$

Concentration Units = Fibers per cubic centimeter

V. The measured concentration is categorized and reported in the following ways:

If The Measured Concentrations Is:	Report As:
1. Less Than the LOD	Less Than LOD
2. Greater Than LOD But Less Than LOQ	Less Than LOQ
3. Greater Than LOQ But Less Than The Minimum Range	Measured Concentration and Recommend Resampling
4. Within The Specified Range	Measured Concentration
5. Greater Than The Specified Range	Measured Concentration and Recommend Resampling

C.11 Quality Assessment Program

In Phase Contrast Microscopy, HASL is involved in 3 quality assessment programs to ensure that HASL's performance in PCM analysis is accurate and precise. The three programs are HASL's intralaboratory program, HASL's interlaboratory program, and the American Industrial Hygiene Association/National Institute For Occupational Health and Safety (AIHA/NIOSH) proficiency in analytical testing (PAT) Program.

1. HASL's intralaboratory program is an ongoing continuous program that includes the following:

1. Daily reference slides analysis for in-house CV
2. Replicate analysis of 10% of samples
3. Duplicate analysis of 10% of samples
4. Laboratory blank analysis of 1 per month (to measure cleanliness of lab)

2. HASL's interlaboratory program is currently a network of independent laboratories conducting PCM and/or asbestos bulk analysis. The network was set up by HASL and all data will be sent to HASL for statistical evaluations. Twice a year, at a given time, all laboratories will send 4 PCM and 4 bulk samples to their assigned laboratories. Each lab is allowed one week to analyze the samples before they must send them to their assigned lab. If there are 8 labs, each lab puts in 4 PCM slides into the analysis chain, by the end of the Round Robin (8 weeks) each lab will have analyzed the 32 samples. There will be a minimum of 2 rounds of PCM and bulk samples per year.

3. The NIST PAT Program consists of a set of samples mailed to the laboratory four times per year. Each analyst must read the samples individually. Only one analyst's results are reported to NIOSH for statistical evaluation (not an average of all results).

C.12 Corrective Actions

When the QA/QC coordinator detects the analyst at the +/- 1 S warning level he/she will resolve with the analyst the problem(s) to bring the analysis back into control. This may include evaluation of the microscope, the analytical procedure, the calculator, anything that may have a bearing on the reported result.

If the problem is with the equipment, an attempt is made to clean and realign the optical system. If the phase resolution slide clearly identify the microscope as the source of the problem, and the QA/QC coordinator has cleaned and realigned the microscope, and the problem persists then the microscope must be sent out for repair.

If the control chart indicates a positive drift in results, then the mounting reagent will be checked for contamination by dropping some reagent onto clean slides and covering the spots with cover glass. If the reagent is the source of the contamination, it should be evident under the microscope.

PART D

POLARIZED LIGHT MICROSCOPY

D.1 PLM Laboratory Operation - General

I. Function:
The use of Polarized Light Microscopy (PLM) is specified in EPA-600/M4-82-020 Method for the Determination of Asbestos in Bulk Insulation Samples. This method was chosen by EPA over other methods because of the quick analysis time, relative low cost, and availability of the instrument.

II. Control:
1. To ensure accurate and precise results, the analytical sequence, equipment, reagents, and analysts must be quality controlled.
2. Materials that might introduce spurious data, should not be introduced into the laboratory.
3. Controls of procedures should be limited to the Laboratory Director and/or his designee and the rules of operation and maintenance should be adhered to strictly.
4. Individual analysts should be thoroughly trained and be under the supervision of an experienced manager.

D.2 Quality Assurance Objectives

The objective of this document is to assure that all data satisfy the EPA interim method with respect to completeness, representativeness and comparability.

In order to meet this objective procedures have to be implemented that strictly control sample receiving, sample preparation, sample analysis, data reduction, reporting of results, record keeping, and performance checking.

D.3 Alignment and Calibration Procedure

A. Stereo Microscope

1. Adjust binocular head so that the field of view appears as one image when looking through with both eyes.
2. Adjust the light source so that the field of view at the lowest magnification (10x) is totally illuminated.
3. Place something onto the microscope to focus on and select a small particle to focus on at 40x. Fine focus the microscope by racking the microscope up or down with either the left or right hand control knob and with the left eye closed.
4. Close the right eye and adjust the Diopter Adjustment (just below the left eyepiece) until the same landmark used in Step 3 is sharp.

B. Polarizing Microscope

1. Dispersion Staining Objective
   i. Place a slide on the rotating stage and focus on any particulate matter.
   ii. Place the dispersion staining lens into the optical system and make certain the slider is not in central or annular stop.
   iii. Place the Bertrand lens out and open the condenser aperture fully.
   iv. Open the field diaphragm until an image of a polygon is about 1/2 the size of the field of view.
   v. Rack the condenser up or down for the sharpest image of the polygon.
   vi. Bring the polygon to the edge of field of view by opening the field diaphragm and center the polygon, if necessary, with the condenser centering screws.
   vii. Open field diaphragm until edge of polygon just disappears.
   viii. Place the Bertrand lens into the optical system.
   ix. Place Dispersion Staining Lens slider into central stop mode and fine center it by adjusting the screw near the slider until the central stop is in center of the field of view.
   x. Focus the Bertrand lens for the sharpest image of the central stop support.
   xi. Close down the aperture on the condenser and make certain the light is centered on the central stop.
   xii. Open the condenser aperture fully and set the attached bottom polar to zero and lock.
   xiii. Adjust the analyzer to obtain the darkest field of view and lock at this position.
   xiv. Slide in 550 millimicron compensator plate and check field of view for characteristic red.
   xv. Place a slide with a particle approximately 5-20 micron in diameter in the center of field of view. Rotate stage and form an imaginary circle

with the particle. With the two rotating stage, centering screws, move the center of the imaginary circle to the center of the field of view. Find another similar sized particle near the center of the field of view and repeat the process. Depending on the skill of the operator this may take only 1-2 attempts or it may take many attempts to center the stage.

2. 10X, 20X, & 40X Objective Lens
   i. Place a slide on the rotating stage and focus on any particulate matter.
   ii. Place the objective lens into the optical system.
   iii. Place the Bertand lens out and open the condenser aperture fully.
   iv. Open the field diaphragm until an image of a polygon is about 1/2 the size of the field of view.
   v. Rack the condenser up or down for the sharpest image of the polygon.
   vi. Bring the polygon to the edge of field of view by opening the field diaphragm and center the polygon, if necessary, with the condenser centering screws.
   vii. Open field diaphragm until edge of polygon just disappears.
   viii. Slide in 550 millimicron compensator plate and check field of view for characteristic red.
   ix. Place a slide with a particle approximately 5-20 micron in diameter in the center of field of view. Rotate stage and form an imaginary circle with the particle. With the two rotating stage, centering screws, move the center of the imaginary circle to the center of the field of view. Find another similar sized particle near the center of the field of view and repeat the process. Depending on the skill of the operator this may take only 1-2 attempts or it may take many attempts to center the stage.

D.4 Equipment Maintenance and Procedures

1. Whenever visible dust is evident on the optical system, it should be cleaned by a gentle spray of clean freon gas and/or with a lint-free tissue. If solvent is indicated, then ethanol applied to a lint-free tissue should be used and care should be exercised not to drip any excess solvent onto any adhesive matrix that may over time loosen part(s) of the optical system.
2. The non-optical parts of the system should be maintained in a state of cleanliness by wiping with a lint-free cloth.
3. When the halogen bulb needs to be replaced, the new bulb should be handled only with a tissue paper - minimizing any transmission of body oil onto the bulb's glass envelope. If oil or other contamination does touch the bulb it will manifest into a shortened bulb life expectancy.
4. If the hood shows any cracking, the stress should be relieved by drilling 1/16" holes at both ends of the defect.
5. HEPA vacuum filters and/or bag should be changed when the airflow into the hood has decreased and the motor is laboring and feels hotter than normal. (See Section D.6 For Procedures)

D.5 Ancillary Equipment and Procedures

I. Hood
The bulk analysis hood must be maintained in a clean state by HEPA-vacuuming followed by wet wiping if needed on an "as needed basis". Small spills can be cleaned by using cellophane tape and allowing the HEPA vac that is used to maintain the negative pressure in the hood, to suck the contaminated cellophane tape into its collection system.

II. HEPA Vacuum
The HEPA vacuum is an electrical filtration device and as such will need maintenance and filter(s) changes. Any such work on the HEPA vac will be performed by qualified repair personnel.

III. Mortar and Pestle
Certain samples need to be physically reduced in size before the analyst can see some of the small fine fibers. Analyst will typically view and feel the sample as they are in their plastic container. If needed, the analyst will reduce the aggregate size by pounding on the exterior of the plastic bag with the mortar and pestle. Holes in the bag (if it occurs) can be easily patched with cellophane tape. The mortar and pestle is cleaned by firmly grasping (if not, the HEPA vac will suck it into the bag) the pestle and place it in the mouth of the vacuum intake of the hood. The mortar is cleaned in a similar fashion.

a. The use of the pestle and mortar is not recommended for samples that contain amphibole minerals or vermiculite. Grinding of amphiboles or vermiculite may result in the separation of fiber bundles or the production of cleavage fragments that have aspect ratios greater than 3:1 and will be classified as asbestos fibers.

IV. Tweezers, Teasing Needles, Scalpels
Tweezers, teasing needles, and scalpels are tools which the analyst uses to manipulate and separate the aggregates into meaningful groupings. The tools will get dirty from manipulating samples and must be HEPA vacuumed by firmly grasping the tool and placing it into the mouth of the vacuum intake of the hood. If this is not done, there will be cross contamination of samples. During the course of analysis, one will dirty one's tools with organic material such as adhesives and asphalt. One cleans organic material off the tools by burning them with a match and scraping off the resultant carbon.

V. Slides and Coverslips .
HASI uses precleaned 1mm thick 75 x 25mm microscope slides and No. 1 1/2 22mm x 22mm cover glasses in the

confirmatory part of the analysis - Polarized Light Microscopy/ Dispersion Staining.

VI. Distilled Water, Dilute HCl, and Dilute Acetic Acid
Warm dilute hydrochloric acid or warm dilute acetic acid may be used to remove calcium carbonate, gypsum, etc.. If acid treatment is required the sample has to be washed at least twice with distilled water. To prevent fiber loss, filtration of the suspension is recommended using MCE filter with pore size of 0.45u (micron) or less.

PREVENTIVE MAINTENANCE CHECKLIST

Item	Frequency	Failure Action
HEPA Vacuum		
HEPA Filters/Bag	When Motor Is Running Hot and Noisy and Airflow Has Decreased	Change Filters and/or Bags
HEPA Vac Inoperative	As Required	Change Carbon Brushes
Hood Defects	As Required	1/16" Holes Drilled At Each End of Defect
Hood		
Hood Vacuum Interior	Weekly	
Wet-Wipe Exterior	Weekly	
Stereo Microscope		
HEPA Vacuum	Weekly	
Oil Movable Parts	As Needed	
Clean Lens	As Needed	
Light Source	As Needed	Change Bulb
PLM/DS Microscope		
Oil Movable Parts	As Needed	
Clean Lens	As Needed	
Light	Each Use	Change Bulb
Wet-Wipe Other Parts	As Needed	
Scalpel	As Needed	Change Blade

D.6 Standard Operating Procedures

General

It is stressed to all HASL personnel that asbestos is a human carcinogen and must be handled with utmost precautions. Any opening of questionable materials must be done inside the hood enclosure with the HEPA vacuum operating. After any manipulation of samples, hands must be washed before answering phones, eating, etc. Any violation of this work rule is a reason for reprimand and continued violations may ultimately result in termination.

The determination of asbestiform minerals involves the experienced and cognizant recognition of several morphologic characteristics and requires the scientific measurement of a series of optical properties. It should be recognized that in characterizing these minerals by dispersion staining, the colors are not absolute since the minerals may be heated, treated, or part of homologous series and will yield varying colors based on crystal chemistry as in actinolite-ferroactinalite-tremolite series.

Difficult samples may require preliminary treatment to separate the fibers from the matrix for examination. The following procedures may be utilized: Mechanical separation (scraping, tweezing, pulverizing, etc.), solvent wash, and wash or ashing. Appropriate precautions should be taken for each treatment to minimize any change in the optical properties of the fibers.

The reported % concentration is evaluated throughout the sample handling process and is the result of an intuitive integration. At low concentrations an available option is the point count method which is a procedure derived from petrographic studies to give estimates of % composition in rocks and is given in the Appendix. HASL also uses the State of New York ELAP's Stratified Point-Counting Technique when appropriate.

Analysis Guidelines:

1. Samples are removed from the outer bag and checked against paperwork on the data sheet.
2. Samples are prepared and analyzed in accordance with EPA 600/M4-82-020 and/or NY ELAP 198.1 or 198.4 depending upon the type and origin of the sample. Techniques used for these analyses are described later in this section. In any case, the number of samples prepared, type of matrix reduction and/or pulverization, refractive index oil used and time spent scanning each slide is at the discretion of the analyst. Each sample is treated separately and care is taken prevent cross contamination.

3. The following are recorded on the analysis data sheet:
   a) Laboratory Identification number.
   b) Stereoscopic Observations:
      - Color
      - Homogeneity
      - Texture
      - Friability
      - Number of fiber types present
      - Number of layers present
      - Tentative identification of fibers
      - Type of sample preparation used.
   c) Polarized Light Microscopic Observations:
      - Asbestos, if present, with the following data:
        - Morphology
        - Extinction
        - Sign of Elongation
        - Birefringence
        - Pleochroism
        - Refractive Index/Dispersion Staining Colors
      - Other fibers present, with the following data:
        - Morphology
        - Sign of Elongation
        - Isotropism, if any
        - Cleavage
        - Relief
        - Birefringence, if any
        - Other identification characteristics
      - Percentage estimation of all material present.
      - Comments on other aspects of the sample.
4. The following are transferred to the HGCL Asbestos Bulk Sample Data Form:
   a) Asbestos type and concentration
   b) Types of other components and concentrations
   c) Color of sample
   d) Type of analysis performed (Point Count, Stratified Point Count, Visual Estimation)
   e) Comments or recommendations

5. The analyst signs the and dates the chain of custody and places the samples into the batch envelope with accompanying paperwork. The samples are then placed in the QC bin.

6. Once in the QC bin, the samples are put through quality control as outlined later in this section. The quality control analyst will initial the QC section of the chain of custody and fax results to the client. (No sample results are reported to the client until the batch has been quality control checked. If the samples are ultra-rush, or there is only one analyst in the office, results must be stamped "Preliminary Results" and faxed to the client. The results may faxed without the stamp only after the batch has been quality control checked.

7. Results are then turned over to the sample log in clerk and entered into the computer system.

Record Keeping:

- Sample data forms are filed by office and by batch number.
- Analysis data sheets are filed numerically by Laboratory sample ID number along with a copy of the associated sample data form(s) stapled to them.
- QC data analysis sheets are filed numerically and by month.

Refractive Index Measurement:

This laboratory uses two methods for measuring refractive index of small particles: the Becke line method and dispersion staining. Both methods are immersion techniques that use Cargille refractive index liquids. Procedures for each method are outlined below.

I. The Becke Line Technique:
Use a filter that transmits yellow light of approximately 589 nm. Choose a refractive index liquid that is most likely to be closely matched to the particle being analyzed. Prepare a dispersion in the chosen oil and choose a typical particle for examination. Set the illumination (substage condenser) for slightly divergent illumination and observe the Becke line (bright line around particle perimeter). When the distance between the objective lens and the object is increased (focus scope up or stage down), the Becke line will move toward the material of higher refraction index: either toward the particle or the oil. Select another oil using the information so obtained and repeat the Becke test. When the fiber is invisible or nearly invisible the refractive index is matched.

II. Dispersion Staining Technique:
Disperse the fibers in the appropriate high dispersion immersions oil and observe the dispersion staining colors when the fiber is oriented in the east-west and north-south directions and record these colors. If the central stop was used find the wavelength that corresponds to the color complement of the dispersion staining color observed. This will be the same color complement of the color observed with the annular filter. Record this wavelength and go to the dispersion curve for the immersion oil used. This curve will show refractive index as a function of wavelength. Locate and record the value of n to which this direction corresponds. Turn the fiber 90o and repeat the procedure. Record the other value of n.

D.7 Other Useful Information

The Effect of Heat On the Microscopical Properties of Asbestos

If the sample has to be dried or burned the following morphological and optical properties changes of asbestos fibers should be considered:

Chrysotile

300^0 C -
- small changes in optical properties
- refractive index decrease slightly
- DS - 1.550 - parallel - blue magenta
  perpendicular - pale blue-green

900^0 C -
- color - light brown
- the fiber bundles develop a granular surface texture and can be very straight
- the fiber bundles become pleochroic
- refractive index increases to approx. 1.635
- negative sign of elongation
- at cross polars the fiber bundles appear polycrystalline

Amosite

300 - 400^0 C
- color - rusty brown
- increased brightness of fibers
- pleochroic - parallel - rusty brown
  - perpendicular - yellow brown
- birefringence - high
- refractive index - increases to approx. 1.747. At higher temperatures the refractive index may increase to 1.95
- sign of elongation remains positive
- the fibers fracture easily crosswise

Crocidolite

300^0 C
- refractive index increases from 1.700 to 1.825
- pleochroism - parallel - red-brown
  perpendicular - yellow-brown
- birefringence - higher than usual
- sign of elongation - changes from negative to positive
- tiny red or orange particles which appear associated with the fibers are iron oxides, a probable by-product of the heating process

Tremolite & Anthophyllite
- very little or no change can be detected

Note: These morphological and optical changes of asbestos fibers due to heating must be considered when analyzing boiler, superheated pipe, and oven insulation.

High Dispersion Oils

When first opened, the refractive index of the dispersion oil shall be confirmed by testing with precision calibrated optical glass. The results must be recorded in the Calibration Book. The oil can then be transferred into smaller bottles. Thereafter, the oil should be retested before bringing it to the hood for use in an analysis.

The oils must also be tested periodically for contamination. This is done by placing a small amount of known non-fibrous material on a slide with a drop of oil and cheking for fibers. If any fibers are detected, the oil should be discarded immediately.

D.8 Quality Control

HASL conducts a minimum of 10 percent duplicate analysis on bulk samples. The samples are chosen at random, and the following criteria are used:

Parameter	Allowable Deviation
Classification	Trace & ND - Acceptable
Identification	Trace & ND - Acceptable
Quantification	Difference - 25% Acceptable

Any difference between the first and second analyst on the above 3 criteria exceeding the allowable deviation, must be recorded in the QC report and the two analyst will work to resolve the difference(s). If the two analysts can not reach a resolution, the Laboratory Manager makes the final decision. All analytical results must be QC before the results are sent out to the client.

In addition to the 10% duplicate analysis, HASL performs selective QC where the QC analyst has the responsibility to check each and every sample that is suspected to be misanalyzed. The samples are chosen at the discretion of the QC analyst and in some instances they are chosen by the or Laboratory Director. In addition, certain matrices which are hard to analyze such as floor tiles and some plasters are "selectively" QC checked more frequently because of their characteristics. All of these results are logged into a book called "Selected QC Results".

To have better control over the analysis and to increase the quality of the work, HASL has instituted a program called Second Opinion, where if an analyst can not make a decision by his/herself, (s)he can ask for a second opinion from another analyst. If both analysts cannot come to an agreement, then the Laboratory Manager has the responsibility to resolve the problem. The final conclusion (result) and comments are recorded in The Second Opinion Book.

Additionally, once a week, the QC Coordinator will give each analyst a blind reference sample to analyze. These reference samples are samples with known types and percentages of asbestos and are from previous rounds of EPA, NIST, ELAP, and NIST's Standard Reference Material 1866 (which consists of chrysotile, amosite, crocidolite, and glass fiber).

Control charts (P chart) based on attributes are plotted to assess the performance of HASL. On the Y-axis are defects per sample and on the x-axis is time (day). A separate chart is maintained for each analyst and for the Laboratory as a unit.

For the purpose of generating control charts for quality assurance, classification and identification will be a single parameter. Quantification will be plotted separately. This grouping of data provides two distinct kinds of information: classification and identification are less subjective then quantification and will be weighted more heavily (except at

very low levels near one percent). Defects as defined above will be plotted for each type and related to specific observers. These data will also be averaged arithmetically to define the laboratory's performance as a unit. The limits of acceptability as defined below will be overlaid on the P-Chart to show an out of bounds condition for which corrective actions are required.

The fraction of defects is plotted as a function of time. The time average of defects is PBAR and is the arithmetic average of defects of each type over the time considered. Out of bounds valves are fractional defects that exceed the upper control limit, UCL, defined by:

UCL (defect) = Expectation (of defect) + 3 x Standard Deviation

At first glance an allowance deviation of 25% for quantification appears to be overly generous, but after reviewing historical EPA/RTI round robin results, 25% is rather conservative.

D.9 Quality Assessment Program

In Polarized Light Microscopy, HASL is involved in 3 quality assessment programs to ensure that HASL's performance in PLM analysis is accurate and precise. The three programs are HASL's intralaboratory program, interlaboratory program, and the Environmental Protection Agency's Asbestos Bulk Sample Analysis Quality Assurance Program.

HASL's intralaboratory program is highlighted in Section D.8 Internal QC Checks along with their statistical equations and explanations and it features an ongoing continuous program of 10% duplicate analysis.

HASL's interlaboratory program is currently a network of independent laboratories conducting PCM and asbestos bulk analysis. Twice a year all laboratories send 2 PCM and 2 bulk samples to their assigned laboratories. Each lab is allowed one week to analyze the samples before they must send them to their assigned lab. If there are 8 labs, each lab puts in 2 Bulk Samples into the analysis chain, by the end of the round robin (8 weeks) each lab will have analyzed the same 16 samples. There will be a minimum of 2 rounds of PCM and bulk samples per year.

PART E

APPENDICES

Appendix 1 - Manual Revision Procedures

I. A Quality Assurance/Quality Control and Procedures Manual is not a static document - it is dynamic and will change as the laboratory upgrades, adds, deletes, and amends, etc. its operational procedures. The change may result because of a change in regulatory procedure or because the Laboratory Director recognizes that certain processes must change to be more efficient, accurate and/or precise.

There are three changes that the Manual can undergo - add, supersede, and delete with some overlaps of the three.

When a new page(s) is added, it is numbered with the original Manual page number that the page(s) is to follow. The new page will also have a suffix of "A" after the original Manual page number to highlight it as an addendum. If there is more than one page then the additional ascending numerical suffix is added.

Example A:	25 - A
Example B:	65 - A - 1.3
	65 - A - 2.3
	65 - A - 3.3

In example A, a single sheet paginated as 25-A was inserted after page 25. In Example B three sheets paginated as 65-A-1.3, 65-A-2.3, and 65-A-3.3 were inserted after page 65. The total sheets of this insert after page 65 is three and this is discerned from the last integer following the decimal point of the last suffix.

If an analytical method is discontinued or is replaced or superseded by another analytical method, the entire section would be superseded by substituting the cover sheet to the method with "Superseded By ". The table of contents would also be similarly amended.

There will be times when a segment of a section must be modified. Documentation of pages deleted and added must be submitted with each change. These documentation of changes will trace the changes of the Manual through the course of its existence as a working document and should remain as part of the Manual. A copy of the QA/QC and procedures manual documentation of changes follows.

It is also possible that excessive changes will make the Manual incomprehensible. If that occurs, HASL will issue a completely new version of its manual along with new ascending numerical version number.

Appendix 2 - Hazardous Waste Manifest

FORM 1: ASBESTOS WASTE MANIFEST (See Instructions on Back)

GENERATOR

1. Work Site Name and Mailing Address | Owner's Name | Owner's Telephone No.

2. Operator's Name and Address | Operator's Telephone No.

3. Waste Disposal Site (WDS) Name, Mailing Address, and Location | WDS Telephone No.

4. Name and Address of Responsible NESHAPS Agency

5. Description of Materials | 6. Containers Number / Type | 7. Total Quantity of Waste (Indicate M^3 or yd^3)

8. Special Handling Instructions and Additional Information

9. OPERATOR'S CERTIFICATION: I hereby declare that the contents of this waste consignment are fully and accurately described above by proper shipping name and are classified, packaged, marked, and labeled; and are in all respects in proper condition for transport by highway according to applicable international and government regulations.

Printed/Typed Name & Title | Signature | Month | Day | Year

TRANSPORTER

10. Transporter 1 (Acknowledgement of Receipt of Materials)

Printed/Typed Name & Title | Signature | Month | Day | Year

Address and Telephone No.

11. Transporter 2 (Acknowledgement of Receipt of Materials)

Printed/Typed Name & Title | Signature | Month | Day | Year

Address and Telephone No.

DISPOSAL SITE

12. Discrepancy Indication Space:

13. Waste Disposal Site Owner or Operator: Certification of receipt of asbestos materials covered by this manifest except as noted in Item 12

Printed/Typed Name & Title | Signature | Month | Day | Year

Project No. _____

Project Manager: _____

INSTRUCTIONS

Waste Generator Section (Items 1-9)

1. Enter the name of the facility at which asbestos waste is generated and the address where the facility is located. In the appropriate spaces, also enter the name of the owner of the facility and the owner's phone number.
2. If a demolition or renovation, enter the name and address of the company and authorized agent responsible for performing the asbestos removal. In the appropriate spaces, also enter the phone number of the operator.
3. Enter the name, address, and physical site location of the waste disposal site (WDS) that will be receiving the asbestos materials. In the appropriate spaces, also enter the phone number of the WDS. Enter "on-site" if the waste will be disposed of on the generator's property.
4. Provide the name and address of the local, State, or EPA Regional Office responsible for administering the asbestos NESHAP program.
5. Indicate the types of asbestos waste materials generated. If from a demolition or renovation, indicate the amount of asbestos that is 1) Friable asbestos material; and 2) Nonfriable asbestos material.
6. Enter the number of containers used to transport the asbestos materials listed in Item 5. Also enter one of the following container codes used in transporting each type of asbestos material (specify any other type of container used if not listed below):

   DM - Metal drums or barrels
   DP - Plastic drums or barrels
   BA - 6 mil plastic bags or wrapping
7. Enter the quantities of each type of asbestos material removed in units of cubic meters (cubic yards).
8. Use this space to indicate special transportation, treatment, storage or disposal, or Bill of Lading information. If an alternate waste disposal site is designated, note it here. Emergency response telephone numbers or similar information may be included here.
9. The authorized agent of the waste generator must read and then sign and date this certification. The date is the date of receipt by transporter.

NOTE: The waste generator must retain a copy of this form.

Transporter Section (Items 10 & 11)

10.& 11. Enter name, address, and telephone number of each transporter used, if applicable. Print or type the full name and title of person accepting responsibility and acknowledging receipt of materials as listed on this waste shipment record for transport. Enter date of receipt and signature.

NOTE: The transporter must retain a copy of this form.

Disposal Site Section (Items 12 & 13)

12. The authorized representative of the WDS must note in this space any discrepancy between waste described on this manifest and waste actually received as well as any improperly enclosed or contained waste. Any rejected materials should be listed and destination of those materials provided. A site that converts asbestos-containing waste material to nonasbestos material is considered a WDS.
13. The signature (by hand) of the authorized WDS agent indicates acceptance and agreement with statements on this manifest except as noted in Item 12. The date is the date of signature and receipt of shipment.

NOTE: The WDS must retain a completed copy of this form. The WDS must also immediately send a completed copy to the operator listed in Item 2.

Appendix 3 - NIOSH 7400 METHOD

FORMULA: various

M.W.: various

FIBERS

METHOD: 7400
ISSUED: 2/15/84
REVISION #3: 5/15/89

PROPERTIES: solid, fibrous

OSHA: 0.2 asbestos fiber (≥5 µm long)/cc;
1 asbestos fiber/cc/30 minute excursion [1]

MSHA: 2 asbestos fibers (>5 µm long)/cc [2]

NIOSH: carcinogen; control to lowest level possible [3]; 3 glass fibers (>10 µm x <3.5 µm)/cc [4]

ACGIH: 0.2 crocidolite; 0.5 amosite; 2 chrysotile and other asbestos, fibers/cc [5]

SYNONYMS: actinolite [CAS #13768-00-8] or ferroactinolite; cummingtonite-grunerite (amosite) [CAS #12172-73-5]; anthophyllite [CAS #17068-78-9]; chrysotile [CAS #12001-29-5] or serpentine; crocidolite [CAS #12001-28-4] or riebeckite; tremolite [CAS #14567-73-8]; amphibole asbestos; fibrous glass.

SAMPLING

SAMPLER: FILTER
(0.45- to 1.2-µm cellulose ester membrane, 25-mm diameter; conductive cowl on cassette)

FLOW RATE*: 0.5 to 16 L/min

VOL-MIN*: 400 L @ 0.1 fiber/cc
-MAX*: (step 4, sampling)

*Adjust to give 100 to 1300 fibers/mm^2

SHIPMENT: routine (pack to reduce shock)

SAMPLE STABILITY: stable

FIELD BLANKS: 10% of samples

ACCURACY

RANGE STUDIED: 80 to 100 fibers counted

BIAS: see EVALUATION OF METHOD

OVERALL PRECISION (s_r): 0.115 to 0.13 [7]

MEASUREMENT

TECHNIQUE: LIGHT MICROSCOPY, PHASE CONTRAST

ANALYTE: fibers (manual count)

SAMPLE PREPARATION: acetone/triacetin "hot block" method [6]

COUNTING RULES: Described in previous version of this method as A rules [1,7]

EQUIPMENT: 1. Positive phase-contrast microscope
2. Walton-Beckett graticule (100-µm field of view) Type G-22
3. phase-shift test slide (HSE/NPL)

CALIBRATION: HSE/NPL test slide

RANGE: 100 to 1300 fibers/mm^2 filter area

ESTIMATED LOD: 7 fibers/mm^2 filter area

PRECISION: 0.10 to 0.12 [7]; see EVALUATION OF METHOD

APPLICABILITY: The quantitative working range is 0.04 to 0.5 fiber/cc for a 1000-L air sample. The LOD depends on sample volume and quantity of interfering dust, and is <0.01 fiber/cc for atmospheres free of interferences. The method gives an index of airborne fibers. It is primarily used for estimating asbestos concentrations, though PCM does not differentiate between asbestos and other fibers. Use this method in conjuction with electron microscopy (e.g., Method 7402) for assistance in identification of fibers. Fiber <ca. 0.25 µm diameter will not be detected by this method [8]. This method may be used for other materials such as fibrous glass by using alternate counting rules (see Appendix C).

INTERFERENCES: Any other airborne fiber may interfere since all particles meeting the counting criteria are counted. Chain-like particles may appear fibrous. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

OTHER METHODS: This method introduces changes for improved sensitivity and reproducibility. It replaces P&CAM 239 [7,9] and NIOSH Method 7400, Revision #2 (dated 8/15/87).

REAGENTS:

1. Acetone.*
2. Triacetin (glycerol triacetate), reagent grade.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler: field monitor, 25-mm, three-piece cassette with ca. 50-mm electrically-conductive extension cowl and cellulose ester filter, 0.45- to 1.2-µm pore size, and backup pad.
   NOTE 1: Analyze representative filters for fiber background before use. Discard the filter lot if mean is ≥5 fibers per 100 graticule fields. These are defined as laboratory blanks. Manufacturer-provided quality assurance checks on filter blanks are normally adequate as long as field blanks are analyzed as described below.
   NOTE 2: The electrically-conductive extension cowl reduces electrostatic effects. Ground the cowl when possible during sampling [10].
   NOTE 3: Use 0.8-µm pore size filters for personal sampling. The 0.45-µm filters are recommended for sampling when performing TEM analysis on the same samples. However, their higher pressure drop precludes their use with personal sampling pumps.
2. Sampling pump, 0.5 to 16 L/min (see step 4 for flow rate), with flexible connecting tubing.
3. Microscope, positive phase (dark) contrast, with green or blue filter, adjustable field iris, 8 to 10X eye-piece, and 40 to 45X phase objective (total magnification ca. 400X); numerical aperture = 0.65 to 0.75.
4. Slides, glass, frosted-end, pre-cleaned, 25- x 75-mm.
5. Cover slips, 22- x 22-mm, No. 1-1/2, unless otherwise specified by microscope manufacturer.
6. Lacquer or nail polish.
7. Knife, #10 surgical steel, curved blade.
8. Tweezers.
9. Heated aluminum block for clearing filters on glass slides (see ref. [6] for specifications or see manufacturer's instructions for equivalent devices).
10. Micropipets, 5-µL and 100- to 500-µL.
11. Graticule, Walton-Beckett type, 100-µm diameter circular field (area = 0.00785 mm^2) at specimen plane (Type G-22). Available from PTR Optics Ltd., 145 Newton Street, Waltham, MA 02154 [phone (617) 891-6000] and McCrone Accessories and Components, 850 Pasquinelli Drive, Westmont, IL 60559 [phone (312) 887-7100].
    NOTE: The graticule is custom-made for each microscope. (See Appendix A for the custom-ordering procedure).
12. HSE/NPL phase contrast test slide, Mark II. Available from PTR Optics Ltd. (address above).
13. Telescope, ocular phase-ring centering.
14. Stage micrometer (0.01-mm divisions).
15. Wire, multi-stranded, 22-gauge.
16. Tape, shrink- or adhesive-.

SPECIAL PRECAUTIONS: Acetone is extremely flammable. Take precautions not to ignite it. Heating of acetone in volumes greater than 1 mL must be done in a ventilated laboratory fume hood using a flameless, spark-free heat source.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. For personal sampling, fasten sampler to the worker's lapel near the worker's mouth. Remove top cover from cowl extension ("open-face") and orient face down. Wrap joint between cowl and monitor body with tape to help hold the cassette together, keep the joint free of dust, and provide a marking surface to identify the cassette.

   NOTE: If possible, ground the cassette to remove any surface charge, using a wire held in contact (e.g., with a hose clamp) with the conductive cowl and an earth ground such as a cold-water pipe.
3. Submit at least two field blanks (or 10% of the total samples, whichever is greater) for each set of samples. Handle field blanks in the same fashion as other samplers. Open field blank cassettes at the same time as other cassettes just prior to sampling. Store top covers and cassettes in a clean area with the top covers from the sampling cassettes during the sampling period.
4. Sample at 0.5 L/min or greater [11]. Adjust sampling flow rate, Q (L/min), and time, t(min), to produce a fiber density, E, of 100 to 1300 fibers/mm^2 ($3.85 \cdot 10^4$ to $5 \cdot 10^5$ fibers per 25-mm filter with effective collection area A_c= 385 mm^2) for optimum accuracy. These variables are related to the action level (one-half the current standard), L (fibers/cc), of the fibrous aerosol being sampled by:

$$t = \frac{A_c \cdot E}{Q \cdot L \cdot 10^3}, \text{ min.}$$

   NOTE 1: The purpose of adjusting sampling times is to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for 8 hrs is appropriate in atmospheres containing ca. 0.1 fiber/cc in the absense of significant amounts of non-asbestos dust. Dusty atmospheres require smaller sample volumes ($\leq$400 L) to obtain countable samples. In such cases take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres, where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3000 to 10000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If $\geq$ 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration.

   NOTE 2: OSHA regulations specify a maximum sampling rate of 2.5 L/min [1].

   NOTE 3: OSHA regulations specify a minimum sampling volume of 48 L for an excursion measurement [1].
5. At the end of sampling, replace top cover and end plugs.
6. Ship samples with conductive cowl attached in a rigid container with packing material to prevent jostling or damage.

   NOTE: Do not use untreated polystyrene foam in shipping container because electrostatic forces may cause fiber loss from sample filter.

SAMPLE PREPARATION:

NOTE 1: The object is to produce samples with a smooth (non-grainy) background in a medium with refractive index of $\leq$1.46. This method collapses the filter for easier focusing and produces relatively permanent mounts which are useful for quality control and interlaboratory comparison. The aluminum "hot block" or similar flash vaporization techniques may be used outside the laboratory [6]. Other mounting techniques meeting the above criteria may also be used (e.g., the laboratory fume hood procedure for generating acetone vapor as described in Method 7400 - revision of 5/15/85, or the non-permanent field mounting technique used in P&CAM 239 [3,7,9,12]). A videotape of the mounting procedure is available from the NIOSH Publication Office [13].

7-91

NOTE 2: Excessive water in the acetone may slow the clearing of the filters, causing material to be washed off the surface of the filter. Also, filters that have been exposed to high humidities prior to clearing may have a grainy background.

7. Ensure that the glass slides and cover slips are free of dust and fibers.
8. Adjust the rheostat to heat the "hot block" to ca. 70 °C [6].
   NOTE: If the "hot block" is not used in a fume hood, it must rest on a ceramic plate and be isolated from any surface susceptible to heat damage.
9. Mount a wedge cut from the sample filter on a clean glass slide.
   a. Cut wedges of ca. 25% of the filter area with a curved-blade knife using a rocking motion to prevent tearing. Place wedge, dust side up, on slide.
      NOTE: Static electricity will usually keep the wedge on the slide.
   b. Insert slide with wedge into the receiving slot at the base of "hot block". Immediately place tip of a micropipet containing ca. 250 µL acetone (use the minimum volume needed to consistently clear the filter sections) into the inlet port of the PTFE cap on top of the "hot block" and inject the acetone into the vaporization chamber with a slow, steady pressure on the plunger button while holding pipet firmly in place. After waiting 3 to 5 sec for the filter to clear, remove pipet and slide from their po·ts.
      CAUTION: Although the volume of acetone used is small, use safety precautions. Work in a well-ventilated area (e.g., laboratory fume hood). Take care not to ignite the acetone. Continuous, frequent use of this device in an unventilated space may produce explosive acetone vapor concentrations.
   c. Using the 5-µL micropipet, immediately place 3.0 to 3.5 µL triacetin on the wedge. Gently lower a clean cover slip onto the wedge at a slight angle to reduce bubble formation. Avoid excess pressure and movement of the cover glass.
      NOTE: If too many bubbles form or the amount of triacetin is insufficient, the cover slip may become detached within a few hours. If excessive triacetin remains at the edge of the filter under the cover slip, fiber migration may occur.
   d. Glue the edges of the cover slip to the slide using lacquer or nail polish [14] Counting may proceed immediately after clearing and mounting are completed.
      NOTE: If clearing is slow, warm the slide on a hotplate (surface temperature 50 °C) for up to 15 min to hasten clearing. Heat carefully to prevent gas bubble formation.

CALIBRATION AND QUALITY CONTROL:

10. Microscope adjustments. Follow the manufacturer's instructions. At least once daily use the telescope ocular (or Bertrand lens, for some microscopes) supplied by the manufacturer to ensure that the phase rings (annular diaphragm and phase-shifting elements) are concentric. With each microscope, keep a logbook in which to record the dates of calibrations and major servicings.
    a. Each time a sample is examined, do the following:
       (1) Adjust the light source for even illumination across the field of view at the condenser iris. Use Kohler illumination, if available. With some microscopes, the illumination may have to be set up with bright field optics rather than phase contrast optics.
       (2) Focus on the particulate material to be examined.
       (3) Make sure that the field iris is in focus, centered on the sample, and open only enough to fully illuminate the field of view.
    b. Check the phase-shift detection limit of the microscope periodically for each analyst/microscope combination:
       (1) Center the HSE/NPL phase-contrast test slide under the phase objective.
       (2) Bring the blocks of grooved lines into focus in the graticule area.
           NOTE: The slide contains seven blocks of grooves (ca. 20 grooves per block) in descending order of visibility. For asbestos counting the microscope optics must completely resolve the grooved lines in block 3 although they may appear somewhat faint, and the grooved lines in blocks 6 and 7 must be invisible

7 / 7

when centered in the graticule area. Blocks 4 or 5 must be at least partially visible but may vary slightly in visibility between microscopes. A microscope which fails to meet these requirements has resolution either too low or too high for fiber counting.

(3) If image quality deteriorates, clean the microscope optics. If the problem persists, consult the microscope manufacturer.

11. Document the laboratory's precision for each counter for replicate fiber counts.
    a. Maintain as part of the laboratory quality assurance program a set of reference slides to be used on a daily basis [15]. These slides should consist of filter preparations including a range of loadings and background dust levels from a variety of sources including both field and PAT samples. The Quality Assurance Officer should maintain custody of the reference slides and should supply each counter with a minimum of one reference slide per workday. Change the labels on the reference slides periodically so that the counter does not become familiar with the samples.
    b. From blind repeat counts on reference slides, estimate the laboratory intra- and intercounter s_r (step 21). Obtain separate values of relative standard deviation for each sample matrix analyzed in each of the following ranges: 5 to 20 fibers in 100 graticule fields, >20 to 50 fibers in 100 graticule fields, >50 to 100 fibers in 100 graticule fields, and 100 fibers in less than 100 graticule fields. Maintain control charts for each of these data files.

    NOTE: Certain sample matrices (e.g., asbestos cement) have been shown to give poor precision [16]

12. Prepare and count field blanks along with the field samples. Report counts on each field blank.

    NOTE 1: The identity of blank filters should be unknown to the counter until all counts have been completed.

    NOTE 2: If a field blank yields greater than 7 fibers per 100 graticule fields, report possible contamination of the samples.

13. Perform blind recounts by the same counter on 10% of filters counted (slides relabeled by a person other than the counter). Use the following test to determine whether a pair of counts by the same counter on the same filter should be rejected because of possible bias: Discard the sample if the absolute value of the difference between the square roots of the two counts (in fiber/mm^2) exceeds 2.8 (X) s_r, where X = the average of the square roots of the two fiber counts (in fiber/mm^2) and s_r = one-half the intracounter relative standard deviation for the appropriate count range (in fibers) determined from step 11. For more complete discussions see reference [15].

    NOTE 1: Since fiber counting is the measurement of randomly placed fibers which may be described by a Poisson distribution, a square root transformation of the fiber count data will result in approximately normally distributed data [15].

    NOTE 2: If a pair of counts is rejected by this test, recount the remaining samples in the set and test the new counts against the first counts. Discard all rejected paired counts. It is not necessary to use this statistic on blank counts.

14. The analyst is a critical part of this analytical procedure. Care must be taken to provide a non-stressful and comfortable environment for fiber counting. An ergonomically designed chair should be used, with the microscope eyepiece situated at a comfortable height for viewing. External lighting should be set at a level similar to the illumination level in the microscope to reduce eye fatigue. In addition, counters should take 10 to 20 minute breaks from the microscope every one or two hours to limit fatigue [17]. During these breaks, both eye and upper back/neck exercises should be performed to relieve strain.

15. All laboratories engaged in asbestos counting should participate in a proficiency testing program such as the AIHA-NIOSH Proficiency Analytical Testing (PAT) Program or the AIHA Asbestos Analyst Registry and routinely exchange field samples with other laboratories to compare performance of counters.

    NOTE: OSHA requires that each analyst performing this method take the NIOSH direct training course #582 or equivalent [1]. Instructors of equivalent courses should have attended the NIOSH #582 course at NIOSH within three years of presenting an equivalent course.

MEASUREMENT:

16. Center the slide on the stage of the calibrated microscope under the objective lens. Focus the microscope on the plane of the filter.
17. Adjust the microscope (Step 10).
    NOTE: Calibration with the HSE/NPL test slide determines the minimum detectable fiber diameter (ca. 0.25 µm) [8].
18. Counting rules: (same as P&CAM 239 rules [3,7,9]; see APPENDIX B).
    a. Count only fibers longer than 5 µm. Measure length of curved fibers along the curve.
    b. Count only fibers with a length-to-width ratio equal to or greater than 3:1.
    c. For fibers which cross the boundary of the graticule field:
       (1) Count any fiber longer than 5 µm which lies entirely within the graticule area.
       (2) Count as 1/2 fiber any fiber with only one end lying within the graticule area, provided that the fiber meets the criteria of rules a and b above.
       (3) Do not count any fiber which crosses the graticule boundary more than once.
       (4) Reject and do not count all other fibers.
    d. Count bundles of fibers as one fiber unless individual fibers can be identified by observing both ends of a fiber.
    e. Count enough graticule fields to yield 100 fibers. Count a minimum of 20 fields. Stop at 100 graticule fields regardless of count.
19. Start counting from the tip of the filter wedge and progress along a radial line to the outer edge. Shift up or down on the filter, and continue in the reverse direction. Select graticule fields randomly by looking away from the eyepiece briefly while advancing the mechanical stage. Ensure that, as a minimum, each analysis covers one radial line from the filter center to the outer edge of the filter. When an agglomerate covers ca. 1/6 or more of the graticule field, reject the graticule field and select another. Do not report rejected graticule fields in the total number counted.

    NOTE 1: When counting a graticule field, continuously scan a range of focal planes by moving the fine focus knob to detect very fine fibers which have become embedded in the filter. The small-diameter fibers will be very faint but are an important contribution to the total count. A minimum counting time of 15 seconds per field is appropriate for accurate counting.

    NOTE 2: This method does not allow for differentiation of fibers based on morphology. Although some experienced counters are capable of selectively counting only fibers which appear to be asbestiform, there is presently no accepted method for ensuring uniformity of judgment between laboratories. It is, therefore, incumbent upon all laboratories using this method to report <u>total</u> fiber counts. If serious contamination from non-asbestos fibers occurs in samples, other techniques such as transmission electron microscopy must be used to identify the asbestos fiber fraction present in the sample (see NIOSH Method 7402). In some cases (i.e., for fibers with diameters >1 µm), polarized light microscopy techniques may be used to identify and eliminate interfering non-crystalline fibers [18].

    NOTE 3: Under certain conditions, electrostatic charge may affect the sampling of fibers. These electrostatic effects are most likely to occur when the relative humidity is low (below 20%), and when sampling is performed near the source of aerosol. The result is that deposition of fibers on the filter is reduced, especially near the edge of the filter. If such a pattern is noted during fiber counting, choose fields as close to the center of the filter as possible [10].

CALCULATIONS AND REPORTING OF RESULTS:

20. Calculate and report fiber density on the filter, E (fibers/mm^2), by dividing the average fiber count per graticule field, F/n_f, minus the mean field blank count per graticule field, B/n_b, by the graticule field area, A_f (approx. 0.00785 mm^2):

$$E = \frac{\left(\frac{F}{n_f} - \frac{B}{n_b}\right)}{A_f} \cdot \text{ fibers/mm}^2.$$

7/16

NOTE: Fiber counts above 1300 fibers/mm^2 and fiber counts from samples with >50% of filter area covered with particulate should be reported as "uncountable" or "probably biased."

21. Calculate and report the concentration, C (fibers/cc), of fibers in the air volume sampled, V (L), using the effective collection area of the filter, A_c (approx. 385 mm^2 for a 25-mm filter):

$$C = \frac{(E)(A_c)}{V \cdot 10^3}.$$

NOTE: Periodically check and adjust the value of A_c, if necessary.

22. Report intralaboratory and interlaboratory relative standard deviations (Step 11) with each set of results.

NOTE: Precision depends on the total number of fibers counted [7,19]. Relative standard deviation is documented in references [7,18,19,20] for fiber counts up to 100 fibers in 100 graticule fields. Comparability of interlaboratory results is discussed below. As a first approximation, use 213% above and 49% below the count as the upper and lower confidence limits for fiber counts greater than 20 (Fig. 1).

EVALUATION OF METHOD:

A. This method is a revision of P&CAM 239 [3,7,9]. A summary of the revisions is as follows:

1. Sampling:
   The change from a 37-mm to a 25-mm filter improves sensitivity for similar air volumes. The change in flow rates allows for 2-m^3 full-shift samples to be taken, providing that the filter is not overloaded with non-fibrous particulates. The collection efficiency of the sampler is not a function of flow rate in the range 0.5 to 16 L/min [11].
2. Sample Preparation Technique:
   The acetone vapor-triacetin preparation technique is a faster, more permanent mounting technique than the dimethyl phthalate/diethyl oxalate method of P&CAM 239 [6,8,9]. The aluminum "hot block" technique minimizes the amount of acetone needed to prepare each sample.
3. Measurement:
   a. The Walton-Beckett graticule standardizes the area observed [21,22,23].
   b. The HSE/NPL test slide standardizes microscope optics for sensitivity to fiber diameter [8,21].
   c. Because of past inaccuracies associated with low fiber counts, the minimum recommended loading has been increased to 100 fibers/mm^2 filter area (80 fibers total count). Lower levels generally result in an overestimate of the fiber count when compared to results in the recommended analytical range [25]. The recommended loadings should yield intracounter s_r in the range of 0.10 to 0.17 [7,24,26].

B. Interlaboratory comparability:
   An international collaborative study involved 16 laboratories using prepared slides from the asbestos cement, milling, mining, textile, and friction material industries [16]. The relative standard deviations (s_r) varied with sample type and laboratory. The ranges were:

	s_r		
	Intralaboratory	Interlaboratory	Overall
AIA (NIOSH Rules)*	0.12 to 0.40	0.27 to 0.85	0.46

*Under AIA rules, only fibers having a diameter less than 3 µm are counted and fibers attached to particles larger than 3 µm are not counted. NIOSH Rules are otherwise similar to the AIA rules.

A NIOSH study was conducted using field samples of asbestos [24]. This study indicated intralaboratory s_r in the range 0.17 to 0.25 and an interlaboratory s_r of 0.45. This agrees well with other recent studies [16,19,21].

At this time, there is no independent means for assessing the overall accuracy of this method. One measure of reliability is to estimate how well the count for a single sample agrees with the mean count from a large number of laboratories. The following discussion indicates how this estimation can be carried out based on measurements of the interlaboratory variability, as well as showing how the results of this method relate to the theoretically attainable counting precision and to measured intra- and interlaboratory s_r. (NOTE: The following discussion does not include bias estimates and should not be taken to indicated that lightly loaded samples are as accurate as properly loaded ones).

Theoretically, the process of counting randomly-distributed (Poisson) fibers on a filter surface will give an s_r that depends on the number, N, of fibers counted:

$$s_r = 1/(N)^{1/2} \qquad (1)$$

Thus s_r is 0.1 for 100 fibers and 0.32 for 10 fibers counted. The actual s_r found in a number of studies is greater than these theoretical numbers [16,19,20,21].

An additional component of variability comes primarily from subjective interlaboratory differences. In a study of ten counters in a continuing sample exchange program, Ogden [18] found this subjective component of intralaboratory s_r to be approximately 0.2 and estimated the overall s_r by the term:

$$\frac{(N + (0.2 \cdot N)^2)^{1/2}}{N} \qquad (2)$$

Ogden found that the 90% confidence interval of the individual intralaboratory counts in relation to the means were +2 s_r and - 1.5 s_r. In this program, one sample out of ten was a quality control sample. For laboratories not engaged in an intensive quality assurance program, the subjective component of variability can be higher.

In a study of field sample results in 46 laboratories, the Asbestos Information Association also found that the variability had both a constant component and one that depended on the fiber count [21]. These results gave a subjective interlaboratory component of s_r (on the same basis as Ogden's) for field samples of ca. 0.45. A similar value was obtained for 12 laboratories analyzing a set of 24 field samples [24]. This value falls slightly above the range of s_r (0.25 to 0.42 for 1984-85) found for 80 reference laboratories in the NIOSH Proficiency Analytical Testing (PAT) program for laboratory-generated samples [20].

A number of factors influence s_r for a given laboratory, such as that laboratory's actual counting performance and the type of samples being analyzed. In the absence of other information, such as from an interlaboratory quality assurance program using field samples, the value for the subjective component of variability is estimated as 0.45. It is hoped that laboratories will carry out the recommended interlaboratory quality assurance programs to improve their performance and thus reduce the s_r.

The above relative standard deviations apply when the population mean has been determined. It is more useful, however, for laboratories to estimate the 90% confidence interval on the mean count from a single sample fiber count (Figure 1). These curves assume similar shapes of the count distribution for interlaboratory and intralaboratory results [19].

For example, if a sample yields a count of 24 fibers, Figure 1 indicates that the mean inter-laboratory count will fall within the range of 227% above and 52% below that value 90% of the time. We can apply these percentages directly to the air concentrations as well. If, for instance, this sample (24 fibers counted) represented a 500-L volume, then the measured concentration is 0.02 fibers/mL (assuming 100 fields counted, 25-mm filter, 0.00785 mm^2 field counting area). If this same sample were counted by a group of laboratories, there is a 90% probability that the mean would fall between 0.01 and 0.08 fiber/mL. These limits should be reported in any comparison of results between laboratories.

Note that the s_r of 0.45 used to derive Figure 1 is used as an estimate for a random group of laboratories. If several laboratories belonging to a quality assurance group can show that their interlaboratory s_r is smaller, then it is more correct to use that smaller s_r. However, the estimated s_r of 0.45 is to be used in the absence of such information. Note also that it has been found that s_r can be higher for certain types of samples, such as asbestos cement [16].

Quite often the estimated airborne concentration from an asbestos analysis is used to compare to a regulatory standard. For instance, if one is trying to show compliance with an 0.5 fiber/mL standard using a single sample on which 100 fibers have been counted, then Figure 1 indicates that the 0.5 fiber/mL standard must be 213% higher than the measured air concentration. This indicates that if one measures a fiber concentration of 0.16 fiber/mL (100 fibers counted), then the mean fiber count by a group of laboratories (of which the compliance laboratory might be one) has a 95% chance of being less than 0.5 fibers/mL; i.e., $0.16 + 2.13 \times 0.16 = 0.5$.

It can be seen from Figure 1 that the Poisson component of the variability is not very important unless the number of fibers counted is small. Therefore, a further approximation is to simply use +213% and -49% as the upper and lower confidence values of the mean for a 100-fiber count.

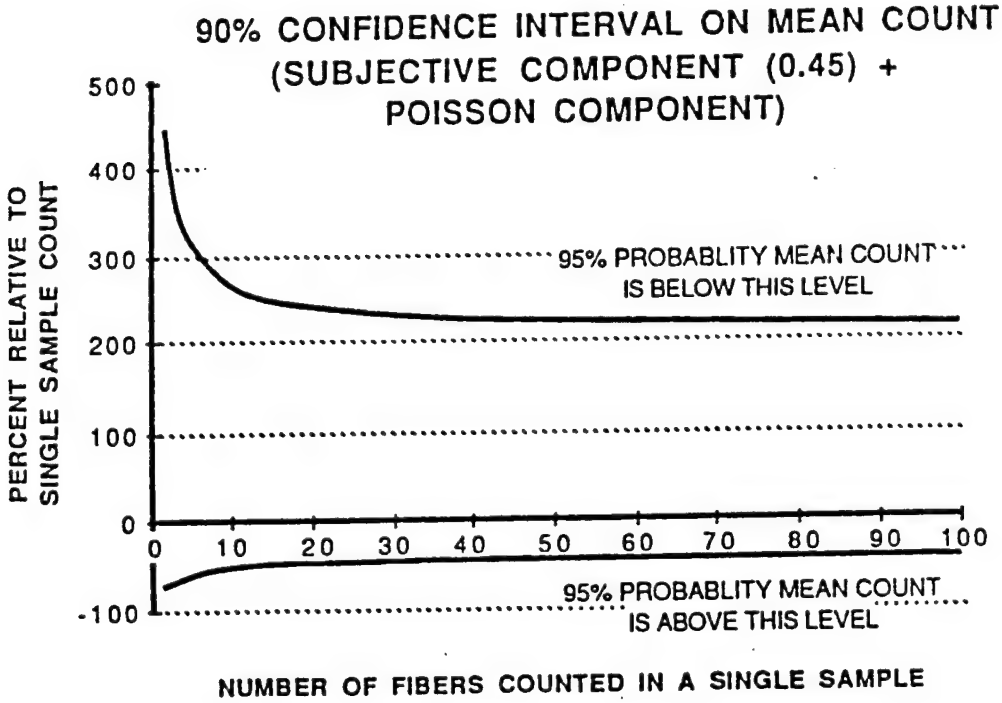


Figure 1. Inter-laboratory Precision of Fiber Counts

The curves in Figure 1 are defined by the following equations

$$UCL = \frac{2x + 2.25 + \sqrt{(2.25 + 2x)^2 - 4(1 - 2.25 s_r^2) x^2}}{2(1 - 2.25 s_r^2)} \quad (3)$$

$$LCL = \frac{2x + 4 - \sqrt{(4 + 2x)^2 - 4(1 - 4 s_r^2) x^2}}{2(1 - 4 s_r^2)} \quad (4)$$

where s_r = subjective inter-laboratory relative standard deviation, which is close to the total inter-laboratory s_r when approximately 100 fibers are counted.

x = total fibers counted on sample

LCL = lower 95% confidence limit

UCL = upper 95% confidence limit.

Note that the range between these two limits represents 90% of the total range.

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APPENDIX A: CALIBRATION OF THE WALTON-BECKETT GRATICULE

Before ordering the Walton-Beckett graticule, the following calibration must be done to obtain a counting area (D) 100 µm in diameter at the image plane. The diameter, d_c (mm), of the circular counting area and the disc diameter must be specified when ordering the graticule.

1. Insert any available graticule into the eyepiece and focus so that the graticule lines are sharp and clear.
2. Set the appropriate interpupillary distance and, if applicable, reset the binocular head adjustment so that the magnification remains constant.
3. Install the 40 to 45X phase objective.
4. Place a stage micrometer on the microscope object stage and focus the microscope on the graduated lines.
5. Measure the magnified grid length of the graticule, L_o (µm), using the stage micrometer.
6. Remove the graticule from the microscope and measure its actual grid length, L_a (mm). This can best be accomplished by using a stage fitted with verniers.
7. Calculate the circle diameter, d_c (mm), for the Walton-Beckett graticule:

$$d_c = \frac{L_a}{L_o} \times D.$$

Example: If L_o = 112 µm, L_a = 4.5 mm and D = 100 µm, then d_c = 4.02 mm.

8. Check the field diameter, D (acceptable range 100 µm ± 2 µm) with a stage micrometer upon receipt of the graticule from the manufacturer. Determine field area (acceptable range 0.00754 to 0.00817 mm^2).

APPENDIX B: EXAMPLES OF COUNTING RULES

Figure 2 shows a Walton-Beckett graticule as seen through the microscope. The rules will be discussed as they apply to the labeled objects in the figure.

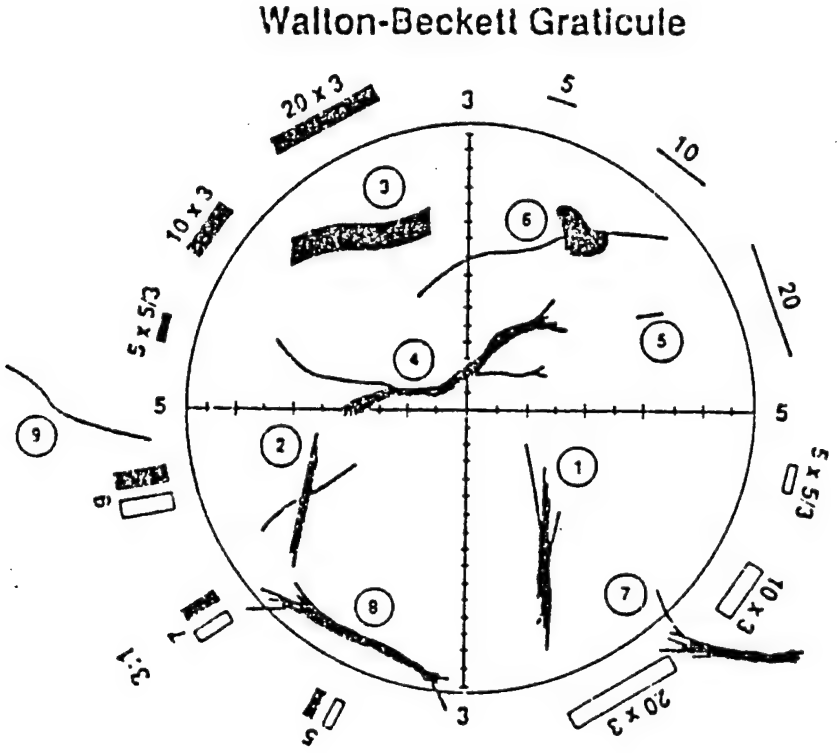

Figure 2. Walton-Beckett graticule with fibers.

7405

FIBER COUNT		
Object	Count	DISCUSSION
1	1 fiber	Optically observable asbestos fibers are actually bundles of fine fibrils. If the fibrils seem to be from the same bundle the object is counted as a single fiber. Note, however, that all objects meeting length and aspect ratio criteria are counted whether or not they appear to be asbestos.
2	2 fiber	If fibers meeting the length and aspect ratio criteria (length >5 µm and length-to-width ratio >3 to 1) overlap, but do not seem to be part of the same bundle, they are counted as separate fibers.
3	1 fiber	Although the object has a relatively large diameter (>3 µm), it is counted as fiber under the rules. There is no upper limit on the fiber diameter in the counting rules. Note that fiber width is measured at the widest compact section of the object.
4	1 fiber	Although long fine fibrils may extend from the body of a fiber, these fibrils are considered part of the fiber if they seem to have originally been part of the bundle.
5	Do not count	If the object is ≤5 µm long, it is not counted.
6	1 fiber	A fiber partially obscured by a particle is counted as one fiber. If the fiber ends emanating from a particle do not seem to be from the same fiber and each end meets the length and aspect ratio criteria, they are counted as separate fibers.
7	1/2 fiber	A fiber which crosses into the graticule area one time is counted as 1/2 fiber.
8	Do not count	Ignore fibers that cross the graticule boundary more than once.
9	Do not count	Ignore fibers that lie outside the graticule boundary.

Appendix C. ALTERNATE COUNTING RULES

Other counting rules may be more appropriate for measurement of specific non-asbestos fiber types, such as fibrous glass. These include the "B" rules (from NIOSH Method 7400, Revision #2, dated 8/15/87), the World Health Organization reference method for man-made mineral fiber [27], and the NIOSH fibrous glass criteria document method [4]. The upper diameter limit in these methods prevents measurements of non-respirable fibers. It is important to note that the aspect ratio limits included in these methods vary. NIOSH recommends the use of the 3:1 aspect ratio in counting fibers.

It is emphasized that hybridization of different sets of counting rules is not permitted. Report specifically which set of counting rules are used with the analytical results.

"B" Counting Rules:

1. Count only ends of fibers. Each fiber must be longer than 5 µm and less than 3 µm diameter.
2. Count only ends of fibers with a length-to-width ratio equal to or greater than 5:1.
3. Count each fiber end which falls within the graticule area as one end, provided that the fiber meets rules b.1 and b.2. Add split ends to the count as appropriate if the split fiber segment also meets the criteria of rules 1 and 2 above.
4. Count visibly free ends which meet rules 1 and 2 above when the fiber appears to be attached to another particle, regardless of the size of the other particle. Count the end of a fiber obscured by another particle if the particle covering the fiber end is less than 3 µm in diameter.
5. Count free ends of fibers emanating from large clumps and bundles up to a maximum of 10 ends (5 fibers), provided that each segment meets rules 1 and 2 above.
6. Count enough graticule fields to yield 200 ends. Count a minimum of 20 graticule fields. Stop at 100 gradicule fields, regardless of count.
7. Divide total end count by 2 to yield fiber count.

Appendix 4 - EPA INTERIM METHOD FOR DETERMINATION OF ASBESTOS IN BULK INSULATION MATERIAL

United States
Environmental Protection
Agency

Environmental Monitoring Systems
Laboratory
Research Triangle Park NC 27711

Research and Development

EPA-600/M4-82-020 Dec. 1982

EPA

Test Method

Interim Method for the Determination of Asbestos in Bulk Insulation Samples*

1. Polarized Light Microscopy

1.1 Principle and Applicability

Bulk samples of building materials taken for asbestos identification are first examined for homogeneity and preliminary fiber identification at low magnification. Positive identification of suspect fibers is made by analysis of subsamples with the polarized light microscope.

The principles of optical mineralogy are well established.[1,2] A light microscope equipped with two polarizing filters is used to observe specific optical characteristics of a sample. The use of plane polarized light allows the determination of refractive indices along specific crystallographic axes Morphology and color are also observed. A retardation plate is placed in the polarized light path for determination of the sign of elongation using orthoscopic illumination. Orientation of the two filters such that their vibration planes are perpendicular (crossed polars) allows observation of the birefringence and extinction characteristics of anisotropic particles.

Quantitative analysis involves the use of point counting. Point counting is a standard technique in petrography for determining the relative areas occupied by separate minerals in thin sections of rock. Background information on the use of point counting[2] and the interpretation of point count data[3] is available.

This method is applicable to all bulk samples of friable insulation materials submitted for identification and quantitation of asbestos components.

1.2 Range

The point counting method may be used for analysis of samples containing from 0 to 100 percent asbestos. The upper detection limit is 100 percent. The lower detection limit is less than 1 percent.

1.3 Interferences

Fibrous organic and inorganic constituents of bulk samples may interfere with the identification and quantitation of the asbestos mineral content. Spray-on binder materials may coat fibers and affect color or obscure optical characteristics to the extent of masking fiber identity. Fine particles of other materials may also adhere to fibers to an extent sufficient to cause confusion in identification. Procedures that may be used for the removal of interferences are presented in Section 1.7.2.2.

1.4 Precision and Accuracy

Adequate data for measuring the accuracy and precision of the method for samples with various matrices are not currently available. Data obtained for samples containing a single asbestos type in a simple matrix are available in the EPA report *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method.*[4]

1.5 Apparatus

1.5.1 Sample Analysis

A low-power binocular microscope, preferably stereoscopic, is used to

*An interim method is carefully drafted from available source information. This method is still under investigation and therefore is subject to revision.

examine the bulk insulation sample as received.

- *Microscope:* binocular, 10-45X (approximate)
- *Light Source:* incandescent or fluorescent
- *Forceps, Dissecting Needles, and Probes*
- *Glassine Paper or Clean Glass Plate*

Compound microscope requirements: A polarized light microscope complete with polarizer, analyzer, port for wave retardation plate, 360° graduated rotating stage, substage condenser, lamp, and lamp iris.

- *Polarized Light Microscope:* described above
- Objective Lenses: 10X, 20X, and 40X or near equivalent
- *Dispersion Staining Objective Lens* (optional)
- *Ocular Lens:* 10X minimum
- *Eyepiece Reticle:* cross hair or 25 point Chalkley Point Array
- *Compensator Plate:* 550 millimicron retardation

1.5.2 Sample Preparation

Sample preparation apparatus requirements will depend upon the type of insulation sample under consideration. Various physical and/or chemical means may be employed for an adequate sample assessment.

- *Ventilated Hood* or negative pressure glove box
- *Microscope Slides*
- *Coverslips*
- *Mortar and Pestle:* agate or porcelain (optional)
- *Wylie Mill* (optional)
- *Beakers & assorted glassware* (optional)
- *Centrifuge* (optional)
- *Filtration apparatus* (optional)
- *Low temperature asher* (optional)

1.6 Reagents

1.6.1 Sample Preparation

- *Distilled Water* (optional)
- *Dilute CH_3COOH:* ACS reagent grade (optional)
- *Dilute HCl:* ACS reagent grade (optional)
- *Sodium metaphosphate* $(NaPO_3)_6$ (optional)

1.6.2 Analytical Reagents

- *Refractive Index Liquids:* 1.490-1.570, 1.590-1.720 in increments of 0.002 or 0.004
- *Refractive Index Liquids for Dispersion Staining:* high-dispersion series, 1.550, 1.605, 1.630 (optional)
- *UICC Asbestos Reference Sample Set:* Available from: UICC MRC Pneumoconiosis Unit, Llandough Hospital, Penarth, Glamorgan CF6 1XW, UK, and commercial distributors
- *Tremolite-asbestos* (source to be determined)
- *Actinolite-asbestos* (source to be determined)

1.7 Procedures

Note: Exposure to airborne asbestos fibers is a health hazard. Bulk samples submitted for analysis are usually friable and may release fibers during handling or matrix reduction steps. All sample and slide preparations should be carried out in a ventilated hood or glove box with continuous airflow (negative pressure). Handling of samples without these precautions may result in exposure of the analyst and contamination of samples by airborne fibers.

1.7.1 Sampling

Samples for analysis of asbestos content shall be taken in the manner prescribed in Reference 5 and information on design of sampling and analysis programs may be found in Reference 6. If there are any questions about the representative nature of the sample, another sample should be requested before proceeding with the analysis.

1.7.2 Analysis

1.7.2.1 Gross Examination

Bulk samples of building materials taken for the identification and quantitation of asbestos are first examined for homogeneity at low magnification with the aid of a stereomicroscope. The core sample may be examined in its container or carefully removed from the container onto a glassine transfer paper or clean glass plate. If possible, note is made of the orientation of top and bottom surfaces. When discrete strata are identified, each is treated as a separate material so that fibers are first identified and quantified in that layer only, and then the results for each layer are combined to yield an estimate of asbestos content for the whole sample.

1.7.2.2 Sample Preparation

Bulk materials submitted for asbestos analysis involve a wide variety of matrix materials. Representative subsamples may not be readily obtainable by simple means in heterogeneous materials, and various steps may be required to alleviate the difficulties encountered. In most cases, however, the best preparation is made by using forceps to sample at several places from the bulk material. Forcep samples are immersed in a refractive index liquid on a microscope slide, teased apart, covered with a cover glass, and observed with the polarized light microscope.

Alternatively, attempts may be made to homogenize the sample or eliminate interferences before further characterization. The selection of appropriate procedures is dependent upon the samples encountered and personal preference. The following are presented as possible sample preparation steps.

A mortar and pestle can sometimes be used in the size reduction of soft or loosely bound materials, though this may cause matting of some samples. Such samples may be reduced in a Wiley mill. Apparatus should be clean and extreme care exercised to avoid cross-contamination of samples. Periodic checks of the particle sizes should be made during the grinding operation so as to preserve any fiber bundles present in an identifiable form. These procedures are not recommended for samples that contain amphibole minerals or vermiculite. Grinding of amphiboles may result in the separation of fiber bundles or the production of cleavage fragments that have aspect ratios greater than 3:1 and will be classified as asbestos fibers. Grinding of vermiculite may also produce fragments with aspect ratios greater than 3:1.

Acid treatment may occasionally be required to eliminate interferences. Calcium carbonate, gypsum, and bassanite (plaster) are frequently present in sprayed or trowelled insulations. These materials may be removed by treatment with warm dilute acetic acid. Warm dilute hydrochloric acid may also be used to remove the above materials. If acid treatment is required, wash the sample at least twice with distilled water, being careful not to lose the particulates during decanting steps. Centrifugation or filtration of the suspension will prevent significant fiber loss. The pore size of the filter should be 0.45 micron or less. *Caution: prolonged acid contact with the sample may alter the optical characteristics of chrysotile fibers and should be avoided.*

Coatings and binding materials adhering to fiber surfaces may also be removed by treatment with sodium metaphosphate.[7] Add 10 mL of 10 g/L sodium metaphosphate solution to a small (0.1 to 0.5 mL) sample of bulk material in a 15-mL glass centrifuge tube. For approximately 15 seconds each, stir the mixture on a vortex mixer, place in an ultrasonic bath and then shake by hand. Repeat the series.

Collect the dispersed solids by centrifugation at 1000 rpm for 5 minutes. Wash the sample three times by suspending in 10 mL distilled water and recentrifuging. After washing, resuspend the pellet in 5 mL distilled water, place a drop of the suspension on a microscope slide, and dry the slide at 110°C.

In samples with a large portion of cellulosic or other organic fibers, it may be useful to ash part of the sample and examine the residue. Ashing should be performed in a low temperature asher. Ashing may also be performed in a muffle furnace at temperatures of 500°C or lower. Temperatures of 550°C or higher will cause dehydroxylation of the asbestos minerals, resulting in changes of the refractive index and other key parameters. If a muffle furnace is to be used, the furnace thermostat should be checked and calibrated to ensure that samples will not be heated at temperatures greater than 500°C.

Ashing and acid treatment of samples should not be used as standard procedures. In order to monitor possible changes in fiber characteristics, the material should be viewed microscopically before and after any sample preparation procedure. Use of these procedures on samples to be used for quantitation requires a correction for percent weight loss.

1.7.2.3 Fiber Identification

Positive identification of asbestos requires the determination of the following optical properties.

- Morphology
- Color and pleochroism
- Refractive indices
- Birefringence
- Extinction characteristics
- Sign of elongation

Table 1-1 lists the above properties for commercial asbestos fibers. Figure 1-1 presents a flow diagram of the examination procedure. Natural variations in the conditions under which deposits of asbestiform minerals are formed will produce exceptions to the published values and differences from the UICC standards. The sign of elongation is determined by use of the compensator plate and crossed polars. Refractive indices may be determined by the Becke line test. Alternatively, dispersion staining may be used. Inexperienced operators may find that the dispersion staining technique is more easily learned, and should consult Reference 9 for guidance. Central stop dispersion staining colors are presented in Table 1-2. Available high-dispersion (HD) liquids should be used.

1.7.2.4 Quantitation of Asbestos Content

Asbestos quantitation is performed by a point-counting procedure. An ocular reticle (cross-hair or point array) is used to visually superimpose a point or points on the microscope field of view. Record the number of points positioned directly above each kind of particle or fiber of interest. Score only points directly over asbestos fibers or nonasbestos matrix material. Do not score empty points for the closest particle. If an asbestos fiber and a matrix particle overlap so that a point is superimposed on their visual intersection, a point is scored for both categories. Point counting provides a determination of the area percent asbestos. Reliable conversion of area percent to percent of dry weight is not currently feasible unless the specific gravities and relative volumes of the materials are known.

For the purpose of this method, "asbestos fibers" are defined as having an aspect ratio greater than 3:1 and being positively identified as one of the minerals in Table 1-1.

A total of 400 points superimposed on either asbestos fibers or nonasbestos matrix material must be counted over at least eight different preparations of representative subsamples. Take eight forcep samples and mount each separately with the appropriate refractive index liquid. The preparation should not be heavily loaded. The sample should be uniformly dispersed to avoid overlapping particles and allow 25-50 percent empty area within the fields of view. Count 50 nonempty points on each preparation, using either

- A cross-hair reticle and mechanical stage; or
- A reticle with 25 points (Chalkley Point Array) and counting at least 2 randomly selected fields.

For samples with mixtures of isotropic and anisotropic materials present, viewing the sample with slightly uncrossed polars or the addition of the compensator plate to the plane polarized light path will allow simultaneous discrimination of both particle types. Quantitation should be performed at 100X or at the lowest magnification of the polarized light microscope that can effectively distinguish the sample components. Confirmation of the quantitation result by a second analyst on some percentage of analyzed samples should be used as standard quality control procedure.

The percent asbestos is calculated as follows:

$$\% \text{ asbestos} = (a/n)\ 100\%$$

where

a = number of asbestos counts,
n = number of nonempty points counted (400).

If $a = 0$, report "No asbestos detected." If $0 < a \leq 3$, report "<1% asbestos."

The value reported should be rounded to the nearest percent.

1.8 References

1. Paul F. Kerr, *Optical Mineralogy,* 4th ed., New York, McGraw-Hill, 1977.
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3. F. Chayes, *Petrographic Modal Analysis: An Elementary Statistical Appraisal,* New York: John Wiley & Sons, 1956.
4. E. P. Brantly, Jr., K. W. Gold, L. E. Myers, and D. E. Lentzen, *Bulk Sample Analysis for Asbestos Content: Evaluation of the Tentative Method,* EPA-600/4-82-021, U.S. Environmental Protection Agency, in preparation.
5. U.S. Environmental Protection Agency, *Asbestos-Containing Materials in School Buildings: A Guidance Document,* Parts 1 and 2, EPA/OTS No. C00090, March 1979.
6. D. Lucas, T. Hartwell, and A. V. Rao, *Asbestos-Containing Materials in School Buildings: Guidance for Asbestos Analytical Programs,* EPA-560/13-80-017A, U.S. Environmental Protection Agency, December 1980.
7. D. H. Taylor and J. S. Bloom, Hexametaphosphate pretreatment of insulation samples for identification of fibrous constituents, *Microscope, 28,* 1980.
8. W. J. Campbell, R. L. Blake, L. L. Brown, E. E. Cather, and J. J. Sjoberg. *Selected Silicate Minerals and Their Asbestiform Varieties: Mineralogical Definitions and Identification-Characterization,* U.S. Bureau of Mines Information Circular 8751, 1977.
9. Walter C. McCrone, *Asbestos Particle Atlas,* Ann Arbor: Ann Arbor Science Publishers, June 1980.

Table 1-1. Optical properties of asbestos fibers

Mineral	Morphology, color[1]	Refractive indices[2] α	γ	Birefringence	Extinction	Sign of elongation
Chrysotile (asbestiform serpentine)	Wavy fibers. Fiber bundles have splayed ends and "kinks". Aspect ratio typically >10:1 Colorless[3], nonpleochroic.	1.493-1.560	1.517-1.562[6] (normally 1.556)	.002-.014	‖to fiber length	+ (length slow)
Amosite (asbestiform grunerite)	Straight, rigid fibers. Aspect ratio typically >10:1. Colorless to brown, nonpleochroic or weakly so. Opaque inclusions may be present.	1.635-1.696	1.655-1.729[6] (normally 1.696-1.710)	.020-.033	‖to fiber length	+ (length slow)
Crocidolite (asbestiform riebeckite)	Straight, rigid fibers. Thick fibers and bundles common, blue to purple-blue in color. Pleochroic. Birefringence is generally masked by blue color.	1.654-1.701	1.668-1.717[5] (normally close to 1.700)	.014-.016	‖to fiber length	— (length fast)
Anthophyllite-asbestos	Straight, single fibers, some larger composite fibers. Anthrophyllite cleavage fragments may be present with aspect ratios < 10:1.[4] Colorless to light brown.	1.596-1.652	1.615-1676[6]	.019-.024	‖to fiber length	+ (length slow)
Tremolite-actinolite-asbestos	Tremolite-asbestos may be present as single or composite fibers. Tremolite cleavage fragments may be present as single crystals with aspect ratios < 10:1.[4] Colorless to pale green.	1.599-1.668	1.622-1.688[6]	.023-.020	Oblique extinction, 10-20° for fragments. Composite fibers show ‖extinction.	+ (length slow)

[1] From reference 5; colors cited are seen by observation with plane polarized light.
[2] From references 5 and 8.
[3] Fibers subjected to heating may be brownish.
[4] Fibers defined as having aspect ratio >3:1.
[5] ⊥ to fiber length.
[6] ‖ to fiber length.

Table 1-2. Central stop dispersion staining colors [a]

Mineral	RI Liquid	⊥	‖
Chrysotile	1.550^{HD}	Blue	Blue-magenta
"Amosite"	1.680	Blue-magenta to pale blue	Golden-yellow
	1.550^{HD}	Yellow to white	Yellow to white
Crocidolite[b]	1.700	Red magenta	Blue-magenta
	1.550^{HD}	Yellow to white	Yellow to white
Anthophyllite-asbestos	1.605^{HD}	Blue	Gold to gold-magenta
Tremolite-asbestos	1.605^{HDc}	Pale blue	Yellow
Actinolite-asbestos	1.605^{HD}	Gold-magenta to blue	Gold
	1.630^{HDc}	Magenta	Golden-yellow

[a] From reference 9, colors may vary slightly.
[b] Blue absorption color.
[c] Oblique extinction view.

Polarized light microscopy qualitative analysis: For each type of material identified by examination of sample at low magnification. Mount spacially dispersed sample in 1.550 RI liquid. (If using dispersion staining, mount in 1.550 HD.) View at 100X with both plane polarized light and crossed polars. More than one fiber type may be present.

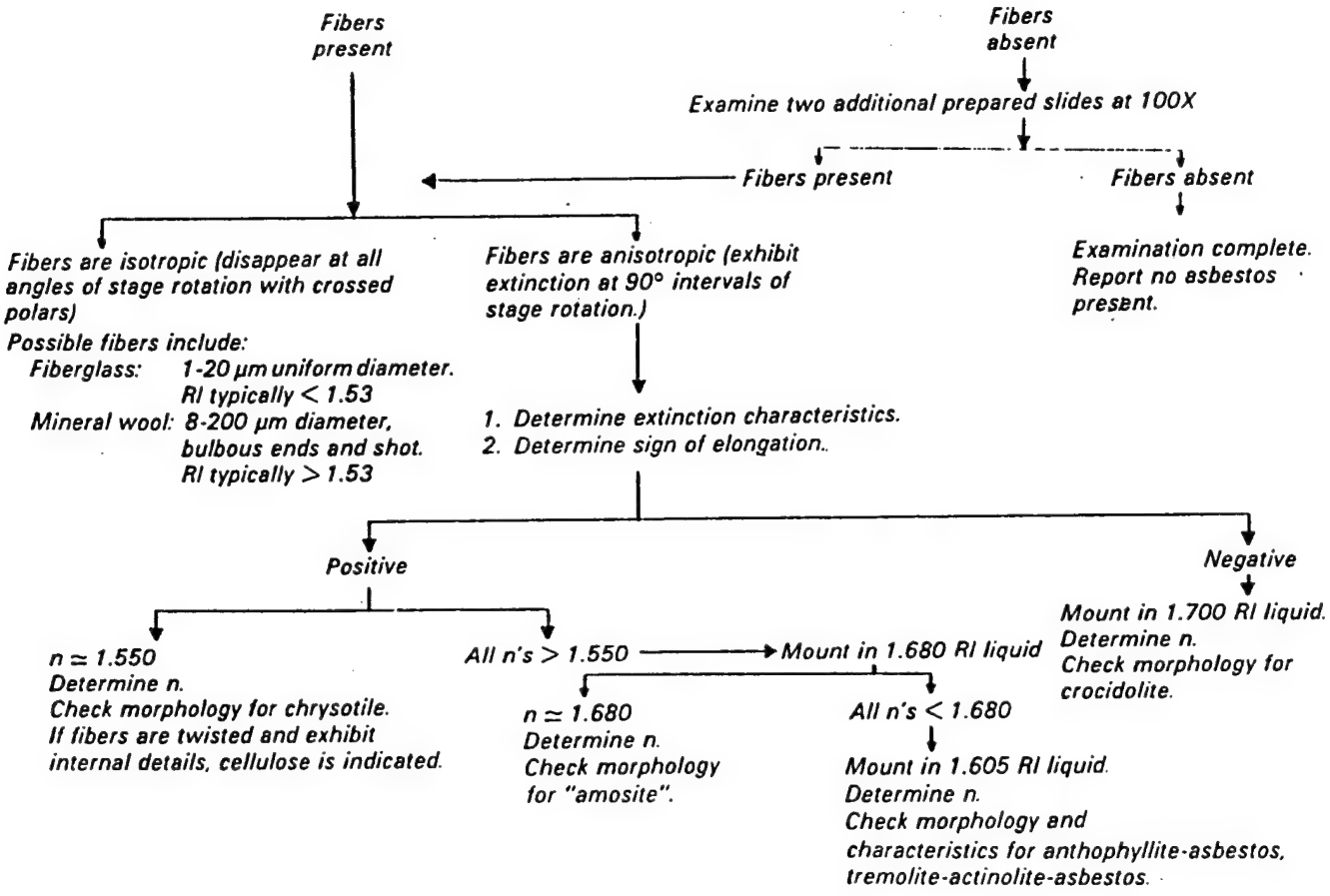
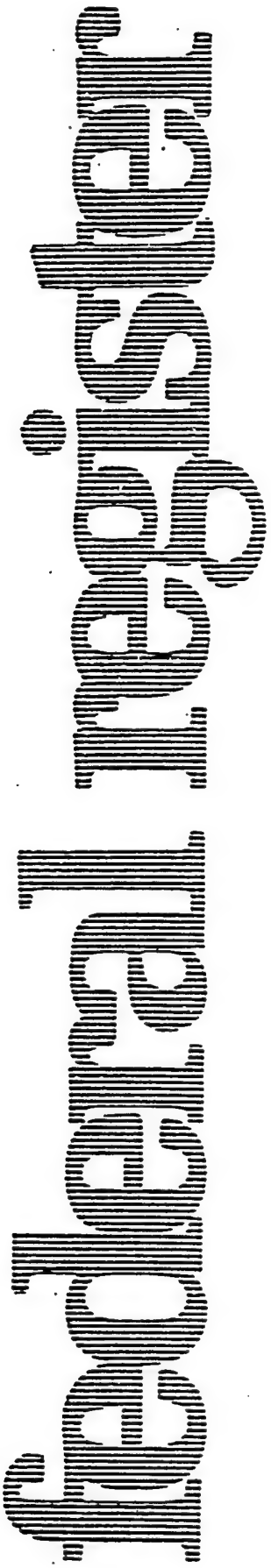


Figure 1-1. Flow chart for qualitative analysis of bulk samples by polarized light microscopy.

Appendix 5 - EPA AHERA REGULATIONS



Friday
October 30, 1987

Part III

Environmental Protection Agency

40 CFR Part 763

Asbestos-Containing Materials In Schools; Final Rule and Notice

program determined to be inadequate, and specifies the facts that underlie the findings of inadequacy.

§ 763.99 Exclusions.

(a) A local education agency shall not be required to perform an inspection under § 763.85(a) in any sampling area as defined in 40 CFR 763.103 or homogeneous area of a school building where:

(1) An accredited inspector has determined that, based on sampling records, friable ACBM was identified in that homogeneous or sampling area during an inspection conducted before December 14, 1987. The inspector shall sign and date a statement to that effect with his or her State of accreditation and if applicable, accreditation number and, within 30 days after such determination, submit a copy of the statement to the person designated under § 763.84 for inclusion in the management plan. However, an accredited inspector shall assess the friable ACBM under § 763.88.

(2) An accredited inspector has determined that, based on sampling records, nonfriable ACBM was identified in that homogeneous or sampling area during an inspection conducted before December 14, 1987. The inspector shall sign and date a statement to that effect with his or her State of accreditation and if applicable, accreditation number and, within 30 days after such determination, submit a copy of the statement to the person designated under § 763.84 for inclusion in the management plan. However, an accredited inspector shall identify whether material that was nonfriable has become friable since that previous inspection and shall assess the newly-friable ACBM under § 763.88.

(3) Based on sampling records and inspection records, an accredited inspector has determined that no ACBM is present in the homogeneous or sampling area and the records show that the area was sampled, before December 14, 1987 in substantial compliance with § 763.85(a), which for purposes of this section means in a random manner and with a sufficient number of samples to reasonably ensure that the area is not ACBM.

(i) The accredited inspector shall sign and date a statement, with his or her State of accreditation and if applicable, accreditation number that the homogeneous or sampling area determined not to be ACBM was sampled in substantial compliance with § 763.85(a).

(ii) Within 30 days after the inspector's determination, the local education agency shall submit a copy of the inspector's statement to the EPA Regional Office and shall include the statement in the management plan for that school.

(4) The lead agency responsible for asbestos inspection in a State that has been granted a waiver from § 763.85(a) has determined that, based on sampling records and inspection records, no ACBM is present in the homogeneous or sampling area and the records show that the area was sampled before December 14, 1987, in substantial compliance with § 763.85(a). Such determination shall be included in the management plan for that school.

(5) An accredited inspector has determined that, based on records of an inspection conducted before December 14, 1987, suspected ACBM identified in that homogeneous or sampling area is assumed to be ACM. The inspector shall sign and date a statement to that effect, with his or her State of accreditation and if applicable, accreditation number and, within 30 days of such determination, submit a copy of the statement to the person designated under § 763.84 for inclusion in the management plan. However, an accredited inspector shall identify whether material that was nonfriable suspected ACBM assumed to be ACM has become friable since the previous inspection and shall assess the newly friable material and previously identified friable suspected ACBM assumed to be ACM under § 763.88.

(6) Based on inspection records and contractor and clearance records, an accredited inspector has determined that no ACBM is present in the homogeneous or sampling area where asbestos removal operations have been conducted before December 14, 1987, and shall sign and date a statement to that effect and include his or her State of accreditation and, if applicable, accreditation number. The local education agency shall submit a copy of the statement to the EPA Regional Office and shall include the statement in the management plan for that school.

(7) An architect or project engineer responsible for the construction of a new school building built after October 12, 1988, or an accredited inspector signs a statement that no ACBM was specified as a building material in any construction document for the building, or, to the best of his or her knowledge, no ACBM was used as a building material in the building. The local education agency shall submit a copy of the signed statement of the architect, project engineer, or accredited inspector to the EPA Regional Office and shall include the statement in the management plan for that school.

(b) The exclusion under paragraph (a)(1) through (4) of this section, from conducting the inspection under § 763.85(a) shall apply only to homogeneous or sampling areas of a school building that were inspected and sampled before October 17, 1987. The local education agency shall conduct an inspection under § 763.85(a) of all areas inspected before October 17, 1987, that were not sampled or were not assumed to be ACM.

(c) If ACBM is subsequently found in a homogeneous or sampling area of a local education agency that had been identified as receiving an exclusion by an accredited inspector under paragraphs (a) (3), (4), (5) of this section, or an architect, project engineer or accredited inspector under paragraph (a)(7) of this section, the local education agency shall have 180 days following the date of identification of ACBM to comply with this Subpart E.

Appendix A to Subpart E—Interim Transmission Electron Microscopy Analytical Methods—Mandatory and Nonmandatory—and Mandatory Section to Determine Completion of Response Actions

I. Introduction

The following appendix contains three units. The first unit is the mandatory transmission electron microscopy (TEM) method which all laboratories must follow; it is the minimum requirement for analysis of air samples for asbestos by TEM. The mandatory method contains the essential elements of the TEM method. The second unit contains the complete non-mandatory method. The non-mandatory method supplements the mandatory method by including additional steps to improve the analysis. EPA recommends that the non-mandatory method be employed for analyzing air filters; however, the laboratory may choose to employ the mandatory method. The non-mandatory method contains the same minimum requirements as are outlined in the mandatory method. Hence, laboratories may choose either of the two methods for analyzing air samples by TEM.

The final unit of this Appendix A to Subpart E defines the steps which must be taken to determine completion of response actions. This unit is mandatory.

II. Mandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. "Analytical sensitivity"—Airborne asbestos concentration represented by each fiber counted under the electron

microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 structures/cm³.

2. "Asbestiform"—A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. "Aspect ratio"—A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. "Bundle"—A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. "Clean area"—A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm² in an area of 0.057 mm² (nominally 10 200-mesh grid openings) and a maximum of 53 structures/mm² for any single preparation for that same area.

6. "Cluster"—A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. "ED"—Electron diffraction.

8. "EDXA"—Energy dispersive X-ray analysis.

9. "Fiber"—A structure greater than or equal to 0.5 µm in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. "Grid"—An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. "Intersection"—Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. "Laboratory sample coordinator"—That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. "Filter background level"—The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on a blank (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm².

14. "Matrix"—Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. "NSD"—No structure detected.

16. "Operator"—A person responsible for the TEM instrumental analysis of the sample.

17. "PCM"—Phase contrast microscopy.

18. "SAED"—Selected area electron diffraction.

19. "SEM"—Scanning electron microscope.

20. "STEM"—Scanning transmission electron microscope.

21. "Structure"—a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. "S/cm³"—Structures per cubic centimeter.

23. "S/mm²"—Structures per square millimeter.

24. "TEM"—Transmission electron microscope.

B. Sampling

1. The sampling agency must have written quality control procedures and documents which verify compliance.

2. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (References 1, 2, 3 and 5 of Unit II.J.).

3. Sampling for airborne asbestos following an abatement action must use commercially available cassettes.

4. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm² in an area of 0.057 mm² (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm² for that same area is acceptable for this method.

5. Use sample collection filters which are either polycarbonate having a pore size less than or equal to 0.4 µm or mixed cellulose ester having a pore size less than or equal to 0.45 µm.

6. Place these filters in series with a 5.0 µm backup filter (to serve as a diffuser) and a support pad. See the following Figure 1:

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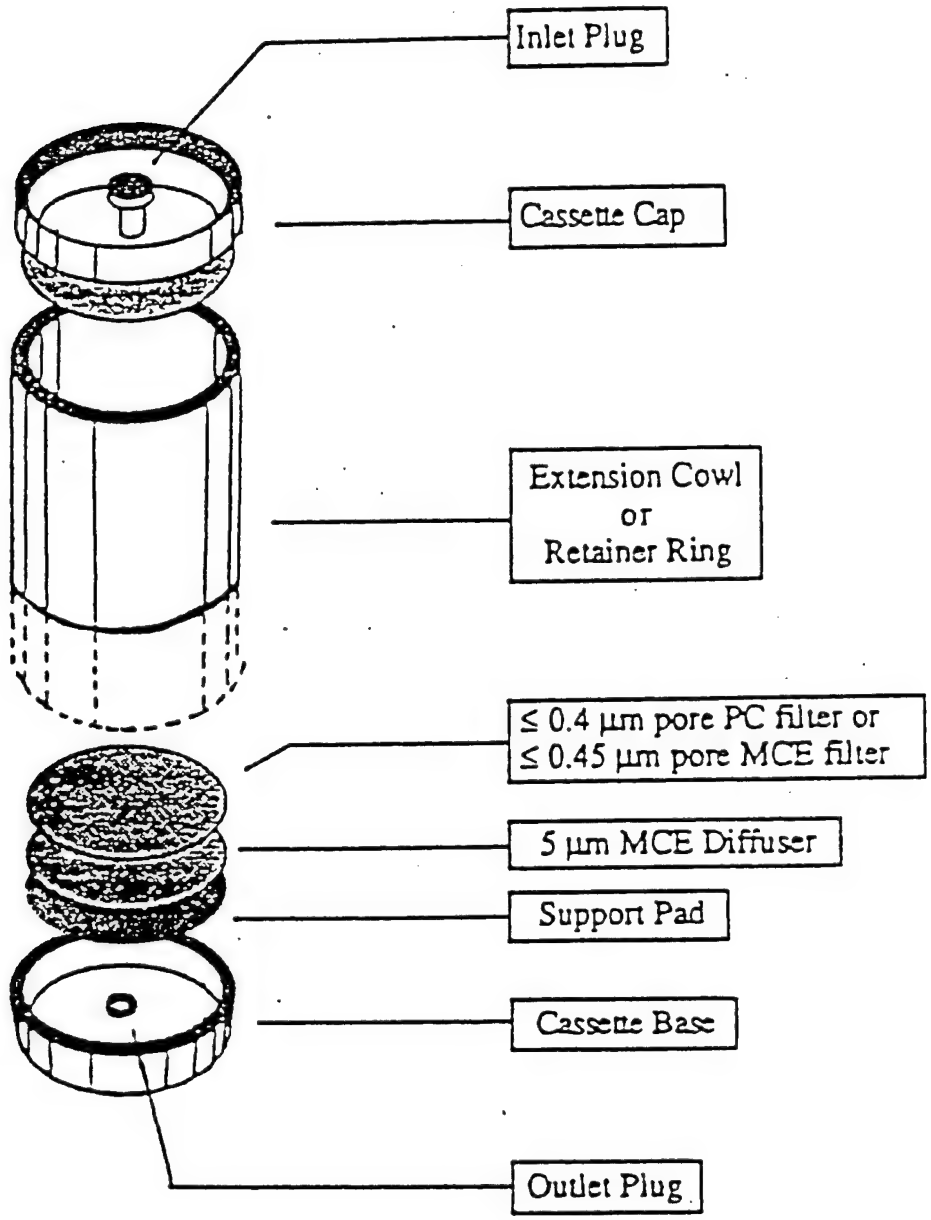
FIGURE I--SAMPLING CASSETTE CONFIGURATION



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7. Reloading of used cassettes is not permitted.

8. Orient the cassette downward at approximately 45 degrees from the horizontal.

9. Maintain a log of all pertinent sampling information.

10. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter (not the filter which will be used in sampling) before and after the sampling operation.

11. Record all calibration information.

12. Ensure that the mechanical vibrations from the pump will be minimized to prevent transferral of vibration to the cassette.

13. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by damping out any pump action fluctuations if necessary.

14. The final plastic barrier around the abatement area remains in place for the sampling period.

15. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust. (See suggested protocol in Unit III.E.7.d.)

16. Select an appropriate flow rate equal to or greater than 1 liter per minute (L/min) or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

17. A minimum of 13 samples are to be collected for each testing site consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 seconds and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each abatement area.

ii. At one of the ambient sites. (DO NOT leave the field blanks open during the sampling period.)

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

18. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

19. The following Table I specifies volume ranges to be used:

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TABLE 1--NUMBER OF 200 MESH EM GRID OPENINGS ($0.0057\ MM^2$) THAT NEED TO BE ANALYZED TO MAINTAIN SENSITIVITY OF 0.005 STRUCTURES/CC BASED ON VOLUME AND EFFECTIVE FILTER AREA

Effective Filter Area 385 sq mm		Effective Filter Area 855 sq mm	
Volume (liters)	# of grid openings	Volume (liters)	# of grid openings
560	*24*	*1,250*	*24*
600	23	1,300	23
700	19	1,400	21
800	17	1,600	19
900	15	1,800	17
1,000	14	2,000	15
1,100	12	2,200	14
1,200	11	2,400	13
1,300	10	2,600	12
1,400	10	2,800	11
1,500	9	3,000	10
1,600	8	3,200	9
1,700	8	3,400	9
1,800	8	3,600	8
1,900	7	3,800	8
2,000	7	4,000	8
2,100	6	4,200	7
2,200	6	4,400	7
2,300	6	4,600	7
2,400	6	4,800	6
2,500	5	5,000	6
2,600	5	5,200	6
2,700	5	5,400	6
2,800	5	5,600	5
2,900	5	5,800	5
3,000	5	6,000	5
3,100	4	6,200	5
3,200	4	6,400	5
3,300	4	6,600	5
3,400	4	6,800	4
3,500	4	7,000	4
3,600	4	7,200	4
3,700	4	7,400	4
3,800	4	7,600	4

Recommended Volume Range

Recommended Volume Range

Note minimum volumes required:
25 mm : 560 liters
37 mm : 1250 liters

Filter diameter of 25 mm = effective area of 385 sq mm
Filter diameter of 37 mm = effective area of 855 sq mm

BILLING CODE 6560-50-C

20. Ensure that the sampler is turned upright before interrupting the pump flow.

21. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

22. Ensure that the samples are stored in a secure and representative location.

23. Do not change containers if portions of these filters are taken for other purposes.

24. A summary of Sample Data Quality Objectives is shown in the following Table II:

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TABLE II--SUMMARY OF SAMPLING AGENCY DATA QUALITY OBJECTIVES

This table summarizes the data quality objectives from the performance of this method in terms of precision, accuracy, completeness, representativeness, and comparability. These objectives are assured by the periodic control checks and reference checks listed here and described in the text of the method.

Unit Operation	QC Check	Frequency	Conformance Expectation
Sampling materials	Sealed blank	1 per I/O site	95%
Sample procedures	Field blanks	2 per I/O site	95%
	Pump calibration	Before and after each field series	90%
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample shipment	Review of sending report	Each sample	95% complete

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C. Sample Shipment

Ship bulk samples to the analytical laboratory in a separate container from air samples.

D. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

E. Sample Preparation

1. All sample preparation and analysis shall be performed by a laboratory independent of the abatement contractor.

2. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them into the clean room facility.

3. Perform sample preparation in a well-equipped clean facility.

Note: The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA-filtered. The cumulative analytical blank concentration must average less than 18 s/mm² in an area of 0.057 mm² (nominally 10 200-mesh grid openings) and a single preparation with a maximum of 53 s/mm² for that same area.

4. Preparation areas for air samples must not only be separated from preparation areas for bulk samples, but they must be prepared in separate rooms.

5. Direct preparation techniques are required. The object is to produce an intact film containing the particulates of the filter surface which is sufficiently clear for TEM analysis.

a. TEM Grid Opening Area measurement must be done as follows:

i. The filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique.

ii. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass and examining it under the PCM. Use a calibrated graticule to measure the average field diameters. From the data, calculate the field area for an average grid opening.

iii. Measurements can also be made on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

b. TEM specimen preparation from polycarbonate (PC) filters. Procedures as described in Unit III.G. or other equivalent methods may be used.

c. TEM specimen preparation from mixed cellulose ester (MCE) filters.

i. Filter portion being used for sample preparation must have the surface collapsed using an acetone vapor technique or the Burdette procedure (Ref. 7 of Unit II.J.)

ii. Plasma etching of the collapsed filter is required. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for the particulate asher and operating conditions will then be set such that a 1–2 μm (10 percent) layer of collapsed surface will be removed.

iii. Procedures as described in Unit III. or other equivalent methods may be used to prepare samples.

F. TEM Method

1. An 80–120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations is required. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely for magnification and camera constant.

2. Determination of Camera Constant and ED Pattern Analysis. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulate. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter of the rings times the interplanar spacing of the ring being measured.

3. Magnification Calibration. The magnification calibration must be done at the fluorescent screen. The TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica (e.g., one containing 2,160 lines/mm). Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric). A logbook must be maintained, and the dates of calibration and the values obtained must be recorded. The frequency of calibration depends on the past history of the particular microscope. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

4. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory.

5. Microscope settings: 80–120 kV, grid assessment 250–1,000X, then 15,000–20,000X screen magnification for analysis.

6. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

7. Individual grid openings with greater than 5 percent openings (holes)

or covered with greater than 25 percent particulate matter or obviously having nonuniform loading must not be analyzed.

8. Reject the grid if:

a. Less than 50 percent of the grid openings covered by the replica are intact.

b. The replica is doubled or folded.

c. The replica is too dark because of incomplete dissolution of the filter.

9. Recording Rules.

a. Any continuous grouping of particles in which an asbestos fiber with an aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 μm is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. An intersection is a nonparallel touching crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. See the following Figure 2:

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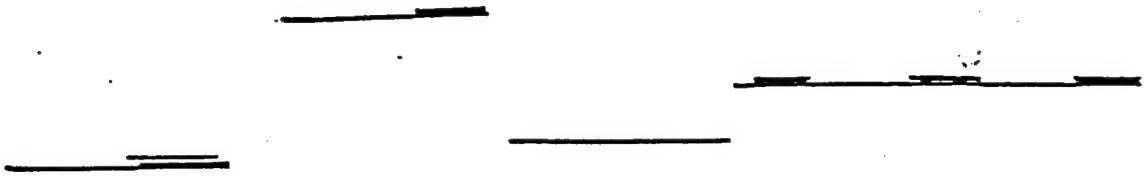
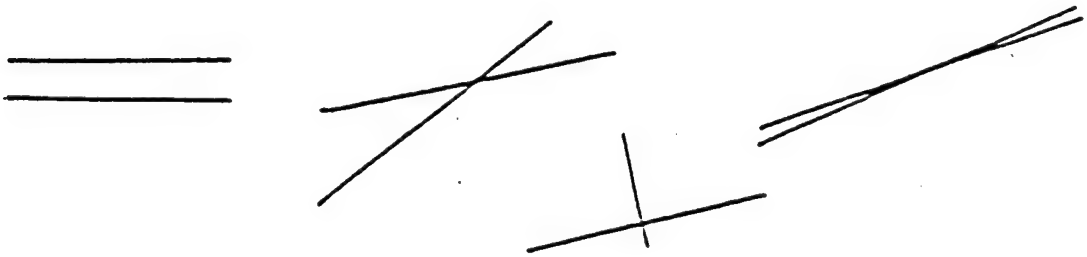
FIGURE 2--COUNTING GUIDELINES USED IN DETERMINING ASBESTOS STRUCTURES

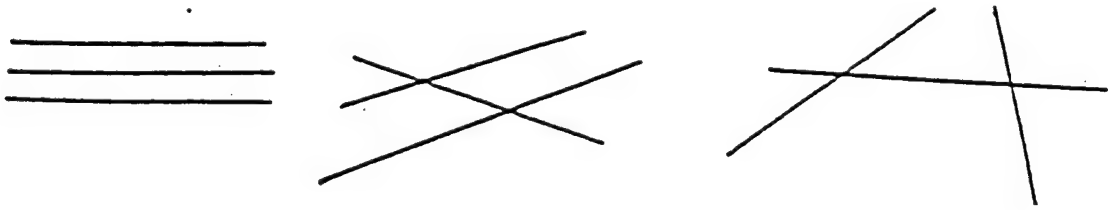
Count as 1 fiber; 1 Structure; no intersections.



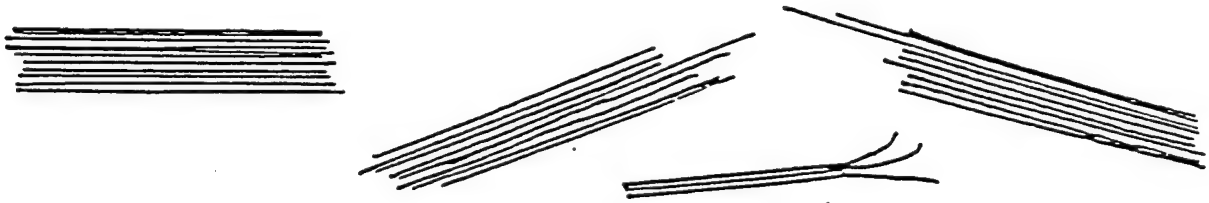
Count as 2 fibers if space between fibers is greater than width of 1 fiber diameter or number of intersections is equal to or less than 1.



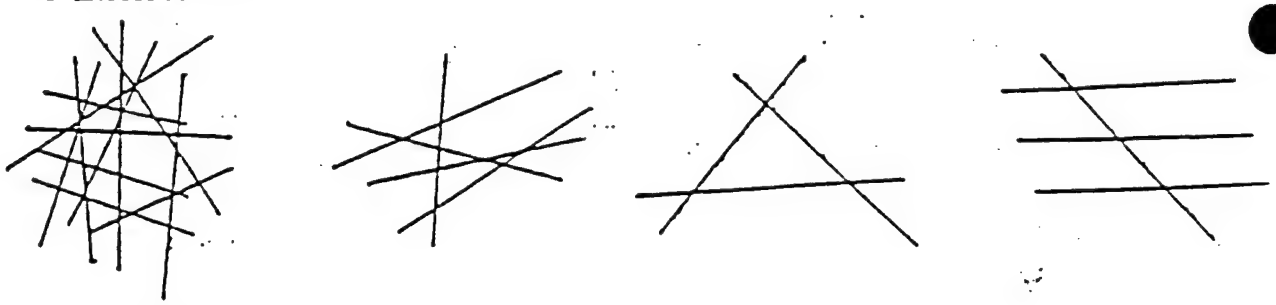
Count as 3 structures if space between fibers is greater than width of 1 fiber diameter or if the number of intersections is equal to or less than 2.



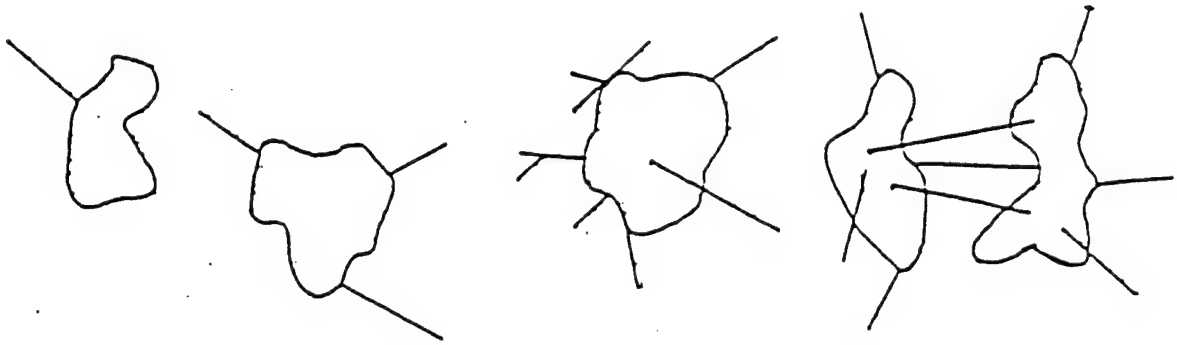
Count bundles as 1 structure; 3 or more parallel fibrils less than 1 fiber diameter separation.



Count clusters as 1 structure; fibers having greater than or equal to 3 intersections.



Count matrix as 1 structure.



DO NOT COUNT AS STRUCTURES:

Fiber protrusion
<5:1 Aspect Ratio

No fiber protusion

Fiber protrusion
<0.5 micrometer

—— <0.5 micrometer in length
— <5:1 Aspect Ratio

BILLING CODE 6560-50-C

i. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle.* A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

iv. *Matrix.* Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

b. Separate categories will be maintained for fibers less than 5 μm and for fibers equal to or greater than 5 μm in length.

c. Record NSD when no structures are detected in the field.

d. Visual identification of electron diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm² concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

e. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. In the event that examination of the pattern by a qualified individual indicates that the pattern has been misidentified visually, the client shall be contacted.

f. Energy Dispersive X-ray Analysis (EDXA) is required of all amphiboles which would cause the analysis results to exceed the 70 s/mm² concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

g. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm² concentration, the fact that they are not asbestos must be confirmed by EDXA or measurement of a zone axis diffraction pattern.

h. Fibers classified as chrysotile must be identified by diffraction or X-ray analysis and recorded on a count sheet. X-ray analysis alone can be used only after 70 s/mm² have been exceeded for a particular sample.

i. Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used only after 70 s/mm² have been exceeded for a particular sample.)

j. If a diffraction pattern was recorded on film, record the micrograph number on the count sheet.

k. If an electron diffraction was attempted but no pattern was observed, record N on the count sheet.

l. If an EDXA spectrum was attempted but not observed, record N on the count sheet.

m. If an X-ray analysis spectrum is stored, record the file and disk number on the count sheet.

10. Classification Rules.

a. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

b. *Bundle.* A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

c. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

d. *Matrix.* Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

11. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid holder. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

C. Sample Analytical Sequence

1. Under the present sampling requirements a minimum of 13 samples is to be collected for the clearance testing of an abatement site. These include five abatement area samples, five ambient samples, two field blanks, and one sealed blank.

2. Carry out visual inspection of work site prior to air monitoring.

3. Collect a minimum of 5 air samples inside the work site and 5 samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

4. Remaining steps in the analytical sequence are contained in Unit IV of this Appendix.

H. Reporting

1. The following information must be reported to the client for each sample analyzed:

a. Concentration in structures per square millimeter and structures per cubic centimeter.

b. Analytical sensitivity used for the analysis.

c. Number of asbestos structures.

d. Area analyzed.

e. Volume of air sampled (which must be initially supplied to lab by client).

f. Copy of the count sheet must be included with the report.

g. Signature of laboratory official to indicate that the laboratory met specifications of the method.

h. Report form must contain official laboratory identification (e.g., letterhead).

i. Type of asbestos.

I. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards are to be performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

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TABLE III--SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or reclean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma etch blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

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1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.

2. Check all laboratory reagents and supplies for acceptable asbestos background levels.

3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks. Testing with blanks must also be done after cleaning or servicing the room.

4. Prepare multiple grids of each sample.

5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If there are more than 53 fibers/mm² per 10 200-mesh grid openings, the system must be checked for possible sources of contamination.

6. Perform a system check on the transmission electron microscope daily.

7. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III under Unit II.I.

8. Ensure qualified operator performance by evaluation of replicate analysis and standard sample comparisons as set forth in Table III under Unit II.I.

9. Validate all data entries.

10. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III under Unit II.I.

11. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the fiber by measurement or comparison of the pattern with patterns collected from standards under the same conditions. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns.

12. Appropriate logs or records must be maintained by the analytical laboratory verifying that it is in compliance with the mandatory quality assurance procedures.

J. References

For additional background information on this method, the following references should be consulted.

1. "Guidance for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5-85-024, June 1985.

2. "Measuring Airborne Asbestos Following an Abatement Action," USEPA, Office of Toxic Substances, EPA 600/4-85-048, 1985.

3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.

4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.

5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4-77-027a, USEPA, Office of Research and Development, 1977.

6. Method 2A: Direct Measurement of Gas Volume through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Burdette, G.J., Health & Safety Exec. Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc., "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters," WERL SOP 87-1, March 5, 1987.

9. NIOSH Method 7402 for Asbestos Fibers, 12-11-86 Draft.

10. Yamate, G., Agarwall, S.C., Gibbons, R.D., IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy," Draft report, USEPA Contract 68-02-3266, July 1984.

11. "Guidance to the Preparation of Quality Assurance Project Plans," USEPA, Office of Toxic Substances, 1984.

III. Nonmandatory Transmission Electron Microscopy Method

A. Definitions of Terms

1. "Analytical sensitivity"—Airborne asbestos concentration represented by each fiber counted under the electron microscope. It is determined by the air volume collected and the proportion of the filter examined. This method requires that the analytical sensitivity be no greater than 0.005 s/cm³.

2. "Asbestiform"—A specific type of mineral fibrosity in which the fibers and fibrils possess high tensile strength and flexibility.

3. "Aspect ratio"—A ratio of the length to the width of a particle. Minimum aspect ratio as defined by this method is equal to or greater than 5:1.

4. "Bundle"—A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

5. "Clean area"—A controlled environment which is maintained and monitored to assure a low probability of asbestos contamination to materials in that space. Clean areas used in this method have HEPA filtered air under positive pressure and are capable of sustained operation with an open laboratory blank which on subsequent analysis has an average of less than 18 structures/mm² in an area of 0.057 mm² (nominally 10 200 mesh grid openings) and a maximum of 53 structures/mm² for no more than one single preparation for that same area.

6. "Cluster"—A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

7. "ED"—Electron diffraction.

8. "EDXA"—Energy dispersive X-ray analysis.

9. "Fiber"—A structure greater than or equal to 0.5 μm in length with an aspect ratio (length to width) of 5:1 or greater and having substantially parallel sides.

10. "Grid"—An open structure for mounting on the sample to aid in its examination in the TEM. The term is used here to denote a 200-mesh copper lattice approximately 3 mm in diameter.

11. "Intersection"—Nonparallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater.

12. "Laboratory sample coordinator"—That person responsible for the conduct of sample handling and the certification of the testing procedures.

13. "Filter background level"—The concentration of structures per square millimeter of filter that is considered indistinguishable from the concentration measured on blanks (filters through which no air has been drawn). For this method the filter background level is defined as 70 structures/mm².

14. "Matrix"—Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

15. "NSD"—No structure detected.

16. "Operator"—A person responsible for the TEM instrumental analysis of the sample.

17. "PCM"—Phase contrast microscopy.

18. "SAED"—Selected area electron diffraction.

19. "SEM"—Scanning electron microscope.

20. "STEM"—Scanning transmission electron microscope.

21. "Structure"—a microscopic bundle, cluster, fiber, or matrix which may contain asbestos.

22. "S/cm³"—Structures per cubic centimeter.

23. "S/mm²"—Structures per square millimeter.

24. "TEM"—Transmission electron microscope.

B. Sampling

1. Sampling operations must be performed by qualified individuals completely independent of the abatement contractor to avoid possible conflict of interest (See References 1, 2, and 5 of Unit III.L.) Special precautions should be taken to avoid contamination of the sample. For example, materials that have not been prescreened for their asbestos background content should not be used; also, sample handling procedures which do not take cross contamination possibilities into account should not be used.

2. Material and supply checks for asbestos contamination should be made on all critical supplies, reagents, and procedures before their use in a monitoring study.

3. Quality control and quality assurance steps are needed to identify problem areas and isolate the cause of the contamination (see Reference 5 of Unit III.L.). Control checks shall be permanently recorded to document the quality of the information produced. The sampling firm must have written quality control procedures and documents which verify compliance. Independent audits by a qualified consultant or firm should be performed once a year. All documentation of compliance should be retained indefinitely to provide a guarantee of quality. A summary of Sample Data Quality Objectives is shown in Table II of Unit II.B.

4. Sampling materials.

a. Sample for airborne asbestos following an abatement action using commercially available cassettes.

b. Use either a cowling or a filter-retaining middle piece. Conductive material may reduce the potential for particulates to adhere to the walls of the cowl.

c. Cassettes must be verified as "clean" prior to use in the field. If packaged filters are used for loading or preloaded cassettes are purchased from the manufacturer or a distributor, the manufacturer's name and lot number should be entered on all field data sheets provided to the laboratory, and are required to be listed on all reports from the laboratory.

d. Assemble the cassettes in a clean facility (See definition of clean area under Unit III.A.).

e. Reloading of used cassettes is not permitted.

f. Use sample collection filters which are either polycarbonate having a pore size of less than or equal to 0.4 μm or mixed cellulose ester having a pore size of less than or equal to 0.45 μm.

g. Place these filters in series with a backup filter with a pore size of 5.0 μm (to serve as a diffuser) and a support pad. See the following Figure 1:

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h. When polycarbonate filters are used, position the highly reflective face such that the incoming particulate is received on this surface.

i. Seal the cassettes to prevent leakage around the filter edges or between cassette part joints. A mechanical press may be useful to achieve a reproducible leak-free seal. Shrink fit gel-bands may be used for this purpose and are available from filter manufacturers and their authorized distributors.

j. Use wrinkle-free loaded cassettes in the sampling operation.

5. Pump setup.

a. Calibrate the sampling pump over the range of flow rates and loads anticipated for the monitoring period with this flow measuring device in series. Perform this calibration using guidance from EPA Method 2A each time the unit is sent to the field (See Reference 6 of Unit III.L.).

b. Configure the sampling system to preclude pump vibrations from being transmitted to the cassette by using a sampling stand separate from the pump station and making connections with flexible tubing.

c. Maintain continuous smooth flow conditions by damping out any pump action fluctuations if necessary.

d. Check the sampling system for leaks with the end cap still in place and the pump operating before initiating sample collection. Trace and stop the source of any flow indicated by the flowmeter under these conditions.

e. Select an appropriate flow rate equal to or greater than 1 L/min or less than 10 L/min for 25 mm cassettes. Larger filters may be operated at proportionally higher flow rates.

f. Orient the cassette downward at approximately 45 degrees from the horizontal.

g. Maintain a log of all pertinent sampling information, such as pump identification number, calibration data, sample location, date, sample identification number, flow rates at the beginning, middle, and end, start and stop times, and other useful information or comments. Use of a sampling log form is recommended. See the following Figure 2:

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FIGURE 2--SAMPLING LOG FORM

Sample Number	Location of Sample	Pump I.D.	Start Time	Middle Time	End Time	Flow Rate

Inspector: _____ Date: _____

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h. Initiate a chain of custody procedure at the start of each sampling, if this is requested by the client.

i. Maintain a close check of all aspects of the sampling operation on a regular basis.

j. Continue sampling until at least the minimum volume is collected, as specified in the following Table 1:

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k. At the conclusion of sampling, turn the cassette upward before stopping the flow to minimize possible particle loss. If the sampling is resumed, restart the flow before reorienting the cassette downward. Note the condition of the filter at the conclusion of sampling.

l. Double check to see that all information has been recorded on the data collection forms and that the cassette is securely closed and appropriately identified using a waterproof label. Protect cassettes in individual clean resealed polyethylene bags. Bags are to be used for storing cassette caps when they are removed for sampling purposes. Caps and plugs should only be removed or replaced using clean hands or clean disposable plastic gloves.

m. Do not change containers if portions of these filters are taken for other purposes.

6. Minimum sample number per site. A minimum of 13 samples are to be collected for each testing consisting of the following:

a. A minimum of five samples per abatement area.

b. A minimum of five samples per ambient area positioned at locations representative of the air entering the abatement site.

c. Two field blanks are to be taken by removing the cap for not more than 30 sec and replacing it at the time of sampling before sampling is initiated at the following places:

i. Near the entrance to each ambient area.

ii. At one of the ambient sites.

[Note: Do not leave the blank open during the sampling period.]

d. A sealed blank is to be carried with each sample set. This representative cassette is not to be opened in the field.

7. Abatement area sampling.

a. Conduct final clearance sampling only after the primary containment barriers have been removed; the abatement area has been thoroughly dried; and, it has passed visual inspection tests by qualified personnel. (See Reference 1 of Unit III.L.)

b. Containment barriers over windows, doors, and air passageways must remain in place until the TEM clearance sampling and analysis is completed and results meet clearance test criteria. The final plastic barrier remains in place for the sampling period.

c. Select sampling sites in the abatement area on a random basis to provide unbiased and representative samples.

d. After the area has passed a thorough visual inspection, use aggressive sampling conditions to dislodge any remaining dust.

i. Equipment used in aggressive sampling such as a leaf blower and/or fan should be properly cleaned and decontaminated before use.

ii. Air filtration units shall remain on during the air monitoring period.

iii. Prior to air monitoring, floors, ceiling and walls shall be swept with the exhaust of a minimum one (1) horsepower leaf blower.

iv. Stationary fans are placed in locations which will not interfere with air monitoring equipment. Fan air is directed toward the ceiling. One fan shall be used for each 10,000 ft ^{3} of worksite.

v. Monitoring of an abatement work area with high-volume pumps and the use of circulating fans will require electrical power. Electrical outlets in the abatement area may be used if available. If no such outlets are available, the equipment must be supplied with electricity by the use of extension cords and strip plug units. All electrical power supply equipment of this type must be approved Underwriter Laboratory equipment that has not been modified. All wiring must be grounded. Ground fault interrupters should be used. Extreme care must be taken to clean up any residual water and ensure that electrical equipment does not become wet while operational.

vi. Low volume pumps may be carefully wrapped in 6-mil polyethylene to insulate the pump from the air. High volume pumps cannot be sealed in this manner since the heat of the motor may melt the plastic. The pump exhausts should be kept free.

vii. If recleaning is necessary, removal of this equipment from the work area must be handled with care. It is not possible to completely decontaminate the pump motor and parts since these areas cannot be wetted. To minimize any problems in this area, all equipment such as fans and pumps should be carefully wet wiped prior to removal from the abatement area. Wrapping and sealing low volume pumps in 6-mil polyethylene will provide easier decontamination of this equipment. Use of clean water and disposable wipes should be available for this purpose.

e. Pump flow rate equal to or greater than 1 L/min or less than 10 L/min may be used for 25 mm cassettes. The larger cassette diameters may have comparably increased flow.

f. Sample a volume of air sufficient to ensure the minimum quantitation limits. (See Table I of Unit III.B.5.j.)

8. Ambient sampling.

a. Position ambient samplers at locations representative of the air entering the abatement site. If makeup air entering the abatement site is drawn from another area of the building which is outside of the abatement area, place the pumps in the building, pumps should be placed out of doors located near the building and away from any obstructions that may influence wind patterns. If construction is in progress immediately outside the enclosure, it may be necessary to select another ambient site. Samples should be representative of any air entering the work site.

b. Locate the ambient samplers at least 3 ft apart and protect them from adverse weather conditions.

c. Sample same volume of air as samples taken inside the abatement site.

C. Sample Shipment

1. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container shall be rejected.

2. Select a rigid shipping container and pack the cassettes upright in a noncontaminating nonfibrous medium such as a bubble pack. The use of resealable polyethylene bags may help to prevent jostling of individual cassettes.

3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.

4. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.

5. Hand-carry samples to the laboratory in an upright position if possible; otherwise choose that mode of transportation least likely to jar the samples in transit.

6. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain of custody and sample tracking procedures. This will also help the laboratory schedule timely analysis for the samples when they are received.

D. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of

sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined, and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the text below.

1. Prescreen the loaded cassette collection filters to assure that they do not contain concentrations of asbestos which may interfere with the analysis of the sample. A filter blank average of less than 18 s/mm² in an area of 0.057 mm² (nominally 10 200-mesh grid openings) and a maximum of 53 s/mm² for that same area for any single preparation is acceptable for this method.

2. Calibrate sampling pumps and their flow indicators over the range of their intended use with a recognized standard. Assemble the sampling system with a representative filter—not the filter which will be used in sampling—before and after the sampling operation.

3. Record all calibration information with the data to be used on a standard sampling form.

4. Ensure that the samples are stored in a secure and representative location.

5. Ensure that mechanical calibrations from the pump will be minimized to prevent transferral of vibration to the cassette.

6. Ensure that a continuous smooth flow of negative pressure is delivered by the pump by installing a damping chamber if necessary.

7. Open a loaded cassette momentarily at one of the indoor sampling sites when sampling is initiated. This sample will serve as an indoor field blank.

8. Open a loaded cassette momentarily at one of the outdoor sampling sites when sampling is initiated. This sample will serve as an outdoor field blank.

9. Carry a sealed blank into the field with each sample series. Do not open this cassette in the field.

10. Perform a leak check of the sampling system at each indoor and outdoor sampling site by activating the pump with the closed sampling cassette in line. Any flow indicates a leak which must be eliminated before initiating the sampling operation.

11. Ensure that the sampler is turned upright before interrupting the pump flow.

12. Check that all samples are clearly labeled and that all pertinent information has been enclosed before transfer of the samples to the laboratory.

E. Sample Receiving

1. Designate one individual as sample coordinator at the laboratory. While that individual will normally be available to receive samples, the coordinator may train and supervise others in receiving procedures for those times when he/she is not available.

2. Adhere to the following procedures to ensure both the continued chain-of-custody and the accountability of all samples passing through the laboratory:

a. Note the condition of the shipping package and data written on it upon receipt.

b. Retain all bills of lading or shipping slips to document the shipper and delivery time.

c. Examine the chain-of-custody seal, if any, and the package for its integrity.

d. If there has been a break in the seal or substantive damage to the package, the sample coordinator shall immediately notify the shipper and a responsible laboratory manager before any action is taken to unpack the shipment.

e. Packages with significant damage shall be accepted only by the responsible laboratory manager after discussions with the client.

3. Unwrap the shipment in a clean, uncluttered facility. The sample coordinator or his or her designee will record the contents, including a description of each item and all identifying numbers or marks. A Sample Receiving Form to document this information is attached for use when necessary. (See the following Figure 3.)

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FIGURE 3--SAMPLE RECEIVING FORM

Date of package delivery _____ Package shipped from _____

Carrier _____ Shipping bill retained _____

*Condition of package on receipt _____

*Condition of custody seal _____

Number of samples received _____ Shipping manifest attached _____

Purchase Order No. _____ Project I.D. _____

Comments _____

No.	Description	Sampling Medium PC	Sampling Medium MCE	Sampled Volume Liters	Receiving ID #	Assigned #
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						

(Use as many additional sheets as needed.)

Comments _____

Date of acceptance into sample bank _____

Signature of chain-of-custody recipient _____

Disposition of samples _____

*Note: If the package has sustained substantial damage or the custody seal is broken, stop and contact the project manager and the shipper.

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Note.—The person breaking the chain-of-custody seal and itemizing the contents assumes responsibility for the shipment and signs documents accordingly.

4. Assign a laboratory number and schedule an analysis sequence.

5. Manage all chain-of-custody samples within the laboratory such that their integrity can be ensured and documented.

F. Sample Preparation

1. Personnel not affiliated with the Abatement Contractor shall be used to prepare samples and conduct TEM analysis. Wet-wipe the exterior of the cassettes to minimize contamination possibilities before taking them to the clean sample preparation facility.

2. Perform sample preparation in a well-equipped clean facility.

Note.—The clean area is required to have the following minimum characteristics. The area or hood must be capable of maintaining a positive pressure with make-up air being HEPA filtered. The cumulative analytical blank concentration must average less than 18 s/mm² in an area of 0.057 s/mm² (nominally 10 200-mesh grid openings) with no more than one single preparation to exceed 53 s/mm² for that same area.

3. Preparation areas for air samples must be separated from preparation areas for bulk samples. Personnel must not prepare air samples if they have previously been preparing bulk samples without performing appropriate personal hygiene procedures, i.e., clothing change, showering, etc.

4. Preparation. Direct preparation techniques are required. The objective is to produce an intact carbon film containing the particulates from the filter surface which is sufficiently clear for TEM analysis. Currently recommended direct preparation procedures for polycarbonate (PC) and mixed cellulose ester (MCE) filters are described in Unit III.F.7. and 8. Sample preparation is a subject requiring additional research. Variation on those steps which do not substantively change the procedure, which improve filter clearing or which reduce contamination problems in a laboratory are permitted.

a. Use only TEM grids that have had grid opening areas measured according to directions in Unit III.J.

b. Remove the inlet and outlet plugs prior to opening the cassette to minimize any pressure differential that may be present.

c. Examples of techniques used to prepare polycarbonate filters are described in Unit III.F.7.

d. Examples of techniques used to prepare mixed cellulose ester filters are described in Unit III.F.8.

e. Prepare multiple grids for each sample.

f. Store the three grids to be measured in appropriately labeled grid holders or polyethylene capsules.

5. Equipment.

a. Clean area.

b. Tweezers. Fine-point tweezers for handling of filters and TEM grids.

c. Scalpel Holder and Curved No. 10 Surgical Blades.

d. Microscope slides.

e. Double-coated adhesive tape.

f. Gummed page reinforcements.

g. Micro-pipet with disposal tips 10 to 100 µL variable volume.

h. Vacuum coating unit with facilities for evaporation of carbon. Use of a liquid nitrogen cold trap above the diffusion pump will minimize the possibility of contamination of the filter surface by oil from the pumping system. The vacuum-coating unit can also be used for deposition of a thin film of gold.

i. Carbon rod electrodes. Spectrochemically pure carbon rods are required for use in the vacuum evaporator for carbon coating of filters.

j. Carbon rod sharpener. This is used to sharpen carbon rods to a neck. The use of necked carbon rods (or equivalent) allows the carbon to be applied to the filters with a minimum of heating.

k. Low-temperature plasma asher. This is used to etch the surface of collapsed mixed cellulose ester (MCE) filters. The asher should be supplied with oxygen, and should be modified as necessary to provide a throttle or bleed valve to control the speed of the vacuum to minimize disturbance of the filter. Some early models of ashers admit air too rapidly, which may disturb particulates on the surface of the filter during the etching step.

l. Glass petri dishes, 10 cm in diameter, 1 cm high. For prevention of excessive evaporation of solvent when these are in use, a good seal must be provided between the base and the lid. The seal can be improved by grinding the base and lid together with an abrasive grinding material.

m. Stainless steel mesh.

n. Lens tissue.

o. Copper 200-mesh TEM grids, 3 mm in diameter, or equivalent.

p. Gold 200-mesh TEM grids, 3 mm in diameter, or equivalent.

q. Condensation washer.

r. Carbon-coated, 200-mesh TEM grids, or equivalent.

s. Analytical balance, 0.1 mg sensitivity.

t. Filter paper, 9 cm in diameter.

u. Oven or slide warmer. Must be capable of maintaining a temperature of 65–70 °C.

v. Polyurethane foam, 6 mm thickness.

w. Gold wire for evaporation.

6. Reagents.

a. General. A supply of ultra-clean, fiber-free water must be available for washing of all components used in the analysis. Water that has been distilled in glass or filtered or deionized water is satisfactory for this purpose. Reagents must be fiber-free.

b. Polycarbonate preparation method—chloroform.

c. Mixed Cellulose Ester (MCE) preparation method—acetone or the Burdette procedure (Ref. 7 of Unit III.L.).

7. TEM specimen preparation from polycarbonate filters.

a. Specimen preparation laboratory. It is most important to ensure that contamination of TEM specimens by extraneous asbestos fibers is minimized during preparation.

b. Cleaning of sample cassettes. Upon receipt at the analytical laboratory and before they are taken into the clean facility or laminar flow hood, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces.

c. Preparation of the carbon evaporator. If the polycarbonate filter has already been carbon-coated prior to receipt, the carbon coating step will be omitted, unless the analyst believes the carbon film is too thin. If there is a need to apply more carbon, the filter will be treated in the same way as an uncoated filter. Carbon coating must be performed with a high-vacuum coating unit. Units that are based on evaporation of carbon filaments in a vacuum generated only by an oil rotary pump have not been evaluated for this application, and must not be used. The carbon rods should be sharpened by a carbon rod sharpener to necks of about 4 mm long and 1 mm in diameter. The rods are installed in the evaporator in such a manner that the points are approximately 10 to 12 cm from the surface of a microscope slide held in the rotating and tilting device.

d. Selection of filter area for carbon coating. Before preparation of the filters, a 75 mm x 50 mm microscope slide is washed and dried. This slide is used to support strips of filter during the carbon evaporation. Two parallel strips of double-sided adhesive tape are applied along the length of the slide. Polycarbonate filters are easily stretched during handling, and cutting of areas for further preparation must be performed with great care. The filter and the MCE backing filter are removed together from the cassette and placed on a cleaned glass microscope slide. The filter can be cut with a curved scalpel blade by rocking the blade from the

point placed in contact with the filter. The process can be repeated to cut a strip approximately 3 mm wide across the diameter of the filter. The strip of polycarbonate filter is separated from the corresponding strip of backing filter and carefully placed so that it bridges the gap between the adhesive tape strips on the microscope slide. The filter strip can be held with fine-point tweezers and supported underneath by the scalpel blade during placement on the microscope slide. The analyst can place several such strips on the same microscope slide, taking care to rinse and wet-wipe the scalpel blade and tweezers before handling a new sample. The filter strips should be identified by etching the glass slide or marking the slide using a marker insoluble in water and solvents. After the filter strip has been cut from each filter, the residual parts of the filter must be returned to the cassette and held in position by reassembly of the cassette. The cassette will then be archived for a period of 30 days or returned to the client upon request.

e. Carbon coating of filter strips. The glass slide holding the filter strips is placed on the rotation-tilting device, and the evaporator chamber is evacuated. The evaporation must be performed in very short bursts, separated by some seconds to allow the electrodes to cool. If evaporation is too rapid, the strips of polycarbonate filter will begin to curl, which will lead to cross-linking of the surface material and make it relatively insoluble in chloroform. An experienced analyst can judge the thickness of carbon film to be applied, and some test should be made first on unused filters. If the film is too thin, large particles will be lost from the TEM specimen, and there will be few complete and undamaged grid openings on the specimen. If the coating is too thick, the filter will tend to curl when exposed to chloroform vapor and the carbon film may not adhere to the support mesh. Too thick a carbon film will also lead to a TEM image that is lacking in contrast and the ability to obtain ED patterns will be compromised. The carbon film should be as thin as possible and remain intact on most of the grid openings of the TEM specimen intact.

f. Preparation of the Jaffe washer. The precise design of the Jaffe washer is not considered important, so any one of the published designs may be used. A washer consisting of a simple stainless steel bridge is recommended. Several pieces of lens tissue approximately 1.0 cm x 0.5 cm are placed on the stainless steel bridge, and the washer is filled with chloroform to a level where the meniscus contacts the underside of the mesh, which results in saturation of the lens tissue. See References 8 and 10 of Unit III.L.

g. Placing of specimens into the Jaffe washer. The TEM grids are first placed on a piece of lens tissue so that individual grids can be picked up with tweezers. Using a curved scalpel blade, the analyst excises three 3 mm square pieces of the carbon-coated polycarbonate filter from the filter strip. The three squares are selected from the center of the strip and from two points between the outer periphery of the active surface and the center. The piece of filter is placed on a TEM specimen grid with the shiny side of the TEM grid facing upwards, and the whole assembly is placed boldly onto the saturated lens tissue in the Jaffe washer. If carbon-coated grids are used, the filter should be placed carbon-coated side down. The three excised squares of filters are placed on the same piece of lens tissue. Any number of separate pieces of lens tissue may be placed in the same Jaffe washer. The lid is then placed on the Jaffe washer, and the system is allowed to stand for several hours, preferably overnight.

h. Condensation washing. It has been found that many polycarbonate filters will not dissolve completely in the Jaffe washer, even after being exposed to chloroform for as long as 3 days. This problem becomes more serious if the surface of the filter was overheated during the carbon evaporation. The presence of undissolved filter medium on the TEM preparation leads to partial or complete obscuration of areas of the sample, and fibers that may be present in these areas of the specimen will be overlooked; this will lead to a low result. Undissolved filter medium also compromises the ability to obtain ED patterns. Before they are counted, TEM grids must be examined critically to determine whether they are adequately cleared of residual filter medium. It has been found that condensation washing of the grids after the initial Jaffe washer treatment, with chloroform as the solvent, clears all residual filter medium in a period of approximately 1 hour. In practice, the piece of lens tissue supporting the specimen grids is transferred to the cold finger of the condensation washer, and the washer is operated for about 1 hour. If the specimens are cleared satisfactorily by the Jaffe washer alone, the condensation washer step may be unnecessary.

8. TEM specimen preparation from MCE filters.

a. This method of preparing TEM specimens from MCE filters is similar to that specified in NIOSH Method 7402. See References 7, 8, and 9 of Unit III.L.

b. Upon receipt at the analytical laboratory, the sample cassettes must be cleaned of any contamination adhering to the outside surfaces before entering the clean sample preparation area.

c. Remove a section from any quadrant of the sample and blank filters.

d. Place the section on a clean microscope slide. Affix the filter section to the slide with a gummed paged reinforcement or other suitable means. Label the slide with a water and solvent-proof marking pen.

e. Place the slide in a petri dish which contains several paper filters soaked with 2 to 3 mL acetone. Cover the dish. Wait 2 to 4 minutes for the sample filter to fuse and clear.

f. Plasma etching of the collapsed filter is required.

i. The microscope slide to which the collapsed filter pieces are attached is placed in a plasma asher. Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. This is one area of the method that requires further evaluation. Insufficient etching will result in a failure to expose embedded filters, and too much etching may result in loss of particulate from the surface. As an interim measure, it is recommended that the time for ashing of a known weight of a collapsed filter be established and that the etching rate be calculated in terms of micrometers per second. The actual etching time used for a particular asher and operating conditions will then be set such that a 1–2 μm (10 percent) layer of collapsed surface will be removed.

ii. Place the slide containing the collapsed filters into a low-temperature plasma asher, and etch the filter.

g. Transfer the slide to a rotating stage inside the bell jar of a vacuum evaporator. Evaporate a 1 mm x 5 mm section of graphite rod onto the cleared filter. Remove the slide to a clean, dry, covered petri dish.

h. Prepare a second petri dish as a Jaffe washer with the wicking substrate prepared from filter or lens paper placed on top of a 6 mm thick disk of clean spongy polyurethane foam. Cut a V-notch on the edge of the foam and filter paper. Use the V-notch as a reservoir for adding solvent. The wicking substrate should be thin enough to fit into the petri dish without touching the lid.

i. Place carbon-coated TEM grids face up on the filter or lens paper. Label the grids by marking with a pencil on the filter paper or by putting registration

marks on the petri dish lid and marking with a waterproof marker on the dish lid. In a fume hood, fill the dish with acetone until the wicking substrate is saturated. The level of acetone should be just high enough to saturate the filter paper without creating puddles.

j. Remove about a quarter section of the carbon-coated filter samples from the glass slides using a surgical knife and tweezers. Carefully place the section of the filter, carbon side down, on the appropriately labeled grid in the acetone-saturated petri dish. When all filter sections have been transferred, slowly add more solvent to the wedge-shaped trough to bring the acetone level up to the highest possible level without disturbing the sample preparations. Cover the petri dish. Elevate one side of the petri dish by placing a slide under it. This allows drops of condensed solvent vapors to form near the edge rather than in the center where they would drip onto the grid preparation.

C. TEM Method

1. Instrumentation.

a. Use an 80–120 kV TEM capable of performing electron diffraction with a fluorescent screen inscribed with calibrated gradations. If the TEM is equipped with EDXA it must either have a STEM attachment or be capable of producing a spot less than 250 nm in diameter at crossover. The microscope shall be calibrated routinely (see Unit III.J.) for magnification and camera constant.

b. While not required on every microscope in the laboratory, the laboratory must have either one microscope equipped with energy dispersive X-ray analysis or access to an equivalent system on a TEM in another laboratory. This must be an Energy Dispersive X-ray Detector mounted on TEM column and associated hardware/software to collect, save, and read out spectral information. Calibration of Multi-Channel Analyzer shall be checked regularly for Al at 1.48 KeV and Cu at 8.04 KeV, as well as the manufacturer's procedures.

i. Standard replica grating may be used to determine magnification (e.g., 2160 lines/mm).

ii. Gold standard may be used to determine camera constant.

c. Use a specimen holder with single tilt and/or double tilt capabilities.

2. Procedure.

a. Start a new Count Sheet for each sample to be analyzed. Record on count sheet: analyst's initials and date; lab sample number; client sample number microscope identification; magnification for analysis; number of predetermined grid openings to be analyzed; and grid identification. See the following Figure 4:

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FIGURE 4--COUNT SHEET

Lab Sample No. _____ Filter Type _____ Operator _____
Client Sample No. _____ Filter Area _____ Date _____
Instrument I.D. _____ Grid I.D. _____ Comments _____
Magnification _____ Grid Opening (GO) Area _____
Acc. Voltage _____ No. GO to be Analyzed _____

GO	Structure No.	Structure Type*	Length		ED Observation				EDAX
			< 5um	≥ 5 um	Chrys.	Amph.	Nonasb.	Ner. ID	

GO	Structure No.	Structure Type*	Length		ED Observation				EDAX
			< 5um	≥ 5 um	Chrys.	Amph.	Nonasb.	Ner. ID	

*B = Bundle
C = Cluster
F = Fiber
M = Matrix

NFD = No fibers detected
N = No diffraction obtained

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b. Check that the microscope is properly aligned and calibrated according to the manufacturer's specifications and instructions.

c. Microscope settings: 80–120 kV, grid assessment 250–1000X, then 15,000–20,000X screen magnification for analysis.

d. Approximately one-half (0.5) of the predetermined sample area to be analyzed shall be performed on one sample grid preparation and the remaining half on a second sample grid preparation.

e. Determine the suitability of the grid.

i. Individual grid openings with greater than 5 percent openings (holes) or covered with greater than 25 percent particulate matter or obviously having nonuniform loading shall not be analyzed.

ii. Examine the grid at low magnification (<1000X) to determine its suitability for detailed study at higher magnifications.

iii. Reject the grid if:

(1) Less than 50 percent of the grid openings covered by the replica are intact.

(2) It is doubled or folded.

(3) It is too dark because of incomplete dissolution of the filter.

iv. If the grid is rejected, load the next sample grid.

v. If the grid is acceptable, continue on to Step 6 if mapping is to be used; otherwise proceed to Step 7.

f. Grid Map (Optional).

i. Set the TEM to the low magnification mode.

ii. Use flat edge or finder grids for mapping.

iii. Index the grid openings (fields) to be counted by marking the acceptable fields for one-half (0.5) of the area needed for analysis on each of the two grids to be analyzed. These may be marked just before examining each grid opening (field), if desired.

iv. Draw in any details which will allow the grid to be properly oriented if it is reloaded into the microscope and a particular field is to be reliably identified.

g. Scan the grid.

i. Select a field to start the examination.

ii. Choose the appropriate magnification (15,000 to 20,000X screen magnification).

iii. Scan the grid as follows.

(1) At the selected magnification, make a series of parallel traverses across the field. On reaching the end of one traverse, move the image one window and reverse the traverse.

Note.—A slight overlap should be used so as not to miss any part of the grid opening (field).

(2) Make parallel traverses until the entire grid opening (field) has been scanned.

h. Identify each structure for appearance and size.

i. Appearance and size: Any continuous grouping of particles in which an asbestos fiber within aspect ratio greater than or equal to 5:1 and a length greater than or equal to 0.5 μm is detected shall be recorded on the count sheet. These will be designated asbestos structures and will be classified as fibers, bundles, clusters, or matrices. Record as individual fibers any contiguous grouping having 0, 1, or 2 definable intersections. Groupings having more than 2 intersections are to be described as cluster or matrix. See the following Figure 5:

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An intersection is a non-parallel touching or crossing of fibers, with the projection having an aspect ratio of 5:1 or greater. Combinations such as a matrix and cluster, matrix and bundle, or bundle and cluster are categorized by the dominant fiber quality—cluster, bundle, and matrix, respectively. Separate categories will be maintained for fibers less than 5 μm and for fibers greater than or equal to 5 μm in length. Not required, but useful, may be to record the fiber length in 1 μm intervals. (Identify each structure morphologically and analyze it as it enters the "window".)

(1) *Fiber.* A structure having a minimum length greater than 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed, no intersections.

(2) *Bundle.* A structure composed of 3 or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

(3) *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group; groupings must have more than 2 intersections.

(4) *Matrix.* Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

(5) *NSD.* Record NSD when no structures are detected in the field.

(6) *Intersection.* Non-parallel touching or crossing of fibers, with the projection having an aspect ratio 5:1 or greater.

ii. Structure Measurement.

(1) Recognize the structure that is to ... sized.

(2) Memorize its location in the "window" relative to the sides, inscribed square and to other particulates in the field so this exact location can be found again when scanning is resumed.

(3) Measure the structure using the scale on the screen.

(4) Record the length category and structure type classification on the count sheet after the field number and fiber number.

(5) Return the fiber to its original location in the window and scan the rest of the field for other fibers; if the direction of travel is not remembered, return to the right side of the field and begin the traverse again.

i. Visual identification of Electron Diffraction (ED) patterns is required for each asbestos structure counted which would cause the analysis to exceed the 70 s/mm² concentration. (Generally this means the first four fibers identified as asbestos must exhibit an identifiable diffraction pattern for chrysotile or amphibole.)

i. Center the structure, focus, and obtain an ED pattern. (See Microscope Instruction Manual for more detailed instructions.)

ii. From a visual examination of the ED pattern, obtained with a short camera length, classify the observed structure as belonging to one of the following classifications: chrysotile, amphibole, or nonasbestos.

(1) Chrysotile: The chrysotile asbestos pattern has characteristic streaks on the layer lines other than the central line and some streaking also on the central line. There will be spots of normal sharpness on the central layer line and on alternate lines (2nd, 4th, etc.). The repeat distance between layer lines is 0.53 nm and the center doublet is at 0.73 nm. The pattern should display (002), (110), (130) diffraction maxima; distances and geometry should match a chrysotile pattern and be measured semiquantitatively.

(2) Amphibole Group (includes grunerite (amosite), crocidolite, anthophyllite, tremolite, and actinolite): Amphibole asbestos fiber patterns show layer lines formed by very closely spaced dots, and the repeat distance between layer lines is also about 0.53 nm. Streaking in layer lines is occasionally present due to crystal structure defects.

(3) Nonasbestos: Incomplete or unobtainable ED patterns, a nonasbestos EDXA, or a nonasbestos morphology.

iii. The micrograph number of the recorded diffraction patterns must be reported to the client and maintained in the laboratory's quality assurance records. The records must also demonstrate that the identification of the pattern has been verified by a qualified individual and that the operator who made the identification is maintaining at least an 80 percent correct visual identification based on his measured patterns. In the event that examination of the pattern by the qualified individual indicates that the pattern had been misidentified visually, the client shall be contacted. If the pattern is a suspected chrysotile, take a photograph of the diffraction pattern at 0 degrees tilt. If the structure is suspected to be amphibole, the sample may have to be tilted to obtain a simple geometric array of spots.

j. Energy Dispersive X-Ray Analysis (EDXA).

i. Required of all amphiboles which would cause the analysis results to exceed the 70 s/mm² concentration. (Generally speaking, the first 4 amphiboles would require EDXA.)

ii. Can be used alone to confirm chrysotile after the 70 s/mm² concentration has been exceeded.

iii. Can be used alone to confirm all nonasbestos.

iv. Compare spectrum profiles with profiles obtained from asbestos standards. The closest match identifies and categorizes the structure.

v. If the EDXA is used for confirmation, record the properly labeled spectrum on a computer disk, or if a hard copy, file with analysis data.

vi. If the number of fibers in the nonasbestos class would cause the analysis to exceed the 70 s/mm² concentration, their identities must be confirmed by EDXA or measurement of a zone axis diffraction pattern to establish that the particles are nonasbestos.

k. Stopping Rules.

i. If more than 50 asbestiform structures are counted in a particular grid opening, the analysis may be terminated.

ii. After having counted 50 asbestiform structures in a minimum of 4 grid openings, the analysis may be terminated. The grid opening in which the 50th fiber was counted must be completed.

iii. For blank samples, the analysis is always continued until 10 grid openings have been analyzed.

iv. In all other samples the analysis shall be continued until an analytical sensitivity of 0.005 s/cm³ is reached.

l. Recording Rules. The count sheet should contain the following information:

i. Field (grid opening): List field number.

ii. Record "NSD" if no structures are detected.

iii. Structure information.

(1) If fibers, bundles, clusters, and/or matrices are found, list them in consecutive numerical order, starting over with each field.

(2) Length. Record length category of asbestos fibers examined. Indicate if less than 5 μm or greater than or equal to 5 μm.

(3) Structure Type. Positive identification of asbestos fibers is required by the method. At least one diffraction pattern of each fiber type from every five samples must be recorded and compared with a standard diffraction pattern. For each asbestos fiber reported, both a morphological descriptor and an identification descriptor shall be specified on the count sheet.

(4) Fibers classified as chrysotile must be identified by diffraction and/or X-ray analysis and recorded on the count

sheet. X-ray analysis alone can be used as sole identification only after 70s/mm² have been exceeded for a particular sample.

(5) Fibers classified as amphiboles must be identified by X-ray analysis and electron diffraction and recorded on the count sheet. (X-ray analysis alone can be used as sole identification only after 70s/mm² have been exceeded for a particular sample.)

(6) If a diffraction pattern was recorded on film, the micrograph number must be indicated on the count sheet.

(7) If an electron diffraction was attempted and an appropriate spectra is not observed, N should be recorded on the count sheet.

(8) If an X-ray analysis is attempted but not observed, N should be recorded on the count sheet.

(9) If an X-ray analysis spectrum is stored, the file and disk number must be recorded on the count sheet.

m. Classification Rules.

i. *Fiber.* A structure having a minimum length greater than or equal to 0.5 μm and an aspect ratio (length to width) of 5:1 or greater and substantially parallel sides. Note the appearance of the end of the fiber, i.e., whether it is flat, rounded or dovetailed.

ii. *Bundle.* A structure composed of three or more fibers in a parallel arrangement with each fiber closer than one fiber diameter.

iii. *Cluster.* A structure with fibers in a random arrangement such that all fibers are intermixed and no single fiber is isolated from the group. Groupings must have more than two intersections.

iv. *Matrix.* Fiber or fibers with one end free and the other end embedded in or hidden by a particulate. The exposed fiber must meet the fiber definition.

v. *NSD.* Record NSD when no structures are detected in the field.

n. After all necessary analyses of a particle structure have been completed, return the goniometer stage to 0 degrees, and return the structure to its original location by recall of the original location.

o. Continue scanning until all the structures are identified, classified and sized in the field.

p. Select additional fields (grid openings) at low magnification; scan at a chosen magnification (15,000 to 20,000X screen magnification); and analyze until the stopping rule becomes applicable.

q. Carefully record all data as they are being collected, and check for accuracy.

r. After finishing with a grid, remove it from the microscope, and replace it in the appropriate grid hold. Sample grids must be stored for a minimum of 1 year from the date of the analysis; the sample cassette must be retained for a minimum of 30 days by the laboratory or returned at the client's request.

H. Sample Analytical Sequence

1. Carry out visual inspection of work site prior to air monitoring.

2. Collect a minimum of five air samples inside the work site and five samples outside the work site. The indoor and outdoor samples shall be taken during the same time period.

3. Analyze the abatement area samples according to this protocol. The analysis must meet the 0.005 s/cm² analytical sensitivity.

4. Remaining steps in the analytical sequence are contained in Unit IV. of this Appendix.

I. Reporting

The following information must be reported to the client. See the following Table II:

BILLING CODE 6560-50-M

TABLE II--EXAMPLE LABORATORY LETTERHEAD

Laboratory I.D.	Client I.D.	FILTER MEDIA DATA				Analyzed Area, mm^2	Sample Volume, cc
		Type	Diameter, mm	Effective Area, mm^2	Pore Size, µm		

INDIVIDUAL ANALYTICAL RESULTS

Laboratory I.D.	Client I.D.	# Asbestos Structures	Analytical Sensitivity, s/cc	CONCENTRATION	
				Structures/mm^2	Structures/cc

The analysis was carried out to the approved TEM method. This laboratory is in compliance with the quality specified by the method.

Authorized Signature

BILLING CODE 6560-50-C

1. Concentration in structures per square millimeter and structures per cubic centimeter.
2. Analytical sensitivity used for the analysis.
3. Number of asbestos structures.
4. Area analyzed.
5. Volume of air samples (which was initially provided by client).
6. Average grid size opening.
7. Number of grids analyzed.
8. Copy of the count sheet must be included with the report.
9. Signature of laboratory official to indicate that the laboratory met specifications of the AHERA method.
10. Report form must contain official laboratory identification (e.g., letterhead).
11. Type of asbestos.

J. Calibration Methodology

Note: Appropriate implementation of the method requires a person knowledgeable in electron diffraction and mineral identification by ED and EDXA. Those inexperienced laboratories wishing to develop capabilities may acquire necessary knowledge through analysis of appropriate standards and by following detailed methods as described in References 8 and 10 of Unit III.L.

1. Equipment Calibration. In this method, calibration is required for the air-sampling equipment and the transmission electron microscope (TEM).

a. TEM Magnification. The magnification at the fluorescent screen of the TEM must be calibrated at the grid opening magnification (if used) and also at the magnification used for fiber counting. This is performed with a cross grating replica. A logbook must be maintained, and the dates of calibration depend on the past history of the particular microscope: no frequency is specified. After any maintenance of the microscope that involved adjustment of the power supplied to the lenses or the high-voltage system or the mechanical disassembly of the electron optical column apart from filament exchange, the magnification must be recalibrated. Before the TEM calibration is performed, the analyst must ensure that the cross grating replica is placed at the same distance from the objective lens as the specimens are. For instruments that incorporate an eucentric tilting specimen stage, all specimens and the cross grating replica must be placed at the eucentric position.

b. Determination of the TEM magnification on the fluorescent screen.

i. Define a field of view on the fluorescent screen either by markings or physical boundaries. The field of view must be measurable or previously inscribed with a scale or concentric circles (all scales should be metric).

ii. Insert a diffraction grating replica (for example a grating containing 2,160 lines/mm) into the specimen holder and place into the microscope. Orient the replica so that the grating lines fall perpendicular to the scale on the TEM fluorescent screen. Ensure that the goniometer stage tilt is 0 degrees.

iii. Adjust microscope magnification to 10,000X or 20,000X. Measure the distance (mm) between two widely separated lines on the grating replica. Note the number of spaces between the lines. Take care to measure between the same relative positions on the lines (e.g., between left edges of lines).

Note.—The more spaces included in the measurement, the more accurate the final calculation. On most microscopes, however, the magnification is substantially constant only within the central 6–10 cm diameter region of the fluorescent screen.

iv. Calculate the true magnification (M) on the fluorescent screen:

$$M = XG/Y$$

where:
X = total distance (mm) between the designated grating lines;
G = calibration constant of the grating replica (lines/mm);
Y = number of grating replica spaces counted along X.

c. Calibration of the EDXA System. Initially, the EDXA system must be calibrated by using two reference elements to calibrate the energy scale of the instrument. When this has been completed in accordance with the manufacturer's instructions, calibration in terms of the different types of asbestos can proceed. The EDXA detectors vary in both solid angle of detection and in window thickness. Therefore, at a particular accelerating voltage in use on the TEM, the count rate obtained from specific dimensions of fiber will vary both in absolute X-ray count rate and in the relative X-ray peak heights for different elements. Only a few minerals are relevant for asbestos abatement work, and in this procedure the calibration is specified in terms of a "fingerprint" technique. The EDXA spectra must be recorded from individual fibers of the relevant minerals, and identifications are made on the basis of semiquantitative comparisons with these reference spectra.

d. Calibration of Grid Openings.

i. Measure 20 grid openings on each of 20 random 200-mesh copper grids by placing a grid on a glass slide and examining it under the PCM. Use a calibrated graticule to measure the average field diameter and use this number to calculate the field area for an average grid opening. Grids are to be randomly selected from batches up to 1,000.

Note.—A grid opening is considered as one field.

ii. The mean grid opening area must be measured for the type of specimen grids in use. This can be accomplished on the TEM at a properly calibrated low magnification or on an optical microscope at a magnification of approximately 400X by using an eyepiece fitted with a scale that has been calibrated against a stage micrometer. Optical microscopy utilizing manual or automated procedures may be used providing instrument calibration can be verified.

e. Determination of Camera Constant and ED Pattern Analysis.

i. The camera length of the TEM in ED operating mode must be calibrated before ED patterns on unknown samples are observed. This can be achieved by using a carbon-coated grid on which a thin film of gold has been sputtered or evaporated. A thin film of gold is evaporated on the specimen TEM grid to obtain zone-axis ED patterns superimposed with a ring pattern from the polycrystalline gold film.

ii. In practice, it is desirable to optimize the thickness of the gold film so that only one or two sharp rings are obtained on the superimposed ED pattern. Thicker gold film would normally give multiple gold rings, but it will tend to mask weaker diffraction spots from the unknown fibrous particulates. Since the unknown d-spacings of most interest in asbestos analysis are those which lie closest to the transmitted beam, multiple gold rings are unnecessary on zone-axis ED patterns. An average camera constant using multiple gold rings can be determined. The camera constant is one-half the diameter, D, of the rings times the interplanar spacing, d, of the ring being measured.

K. Quality Control/Quality Assurance Procedures (Data Quality Indicators)

Monitoring the environment for airborne asbestos requires the use of sensitive sampling and analysis procedures. Because the test is sensitive, it may be influenced by a variety of factors. These include the supplies used in the sampling operation, the performance of the sampling, the preparation of the grid from the filter and the actual examination of this grid in the microscope. Each of these unit operations must produce a product of

defined quality if the analytical result is to be a reliable and meaningful test result. Accordingly, a series of control checks and reference standards is performed along with the sample analysis as indicators that the materials used are adequate and the operations are within acceptable limits. In this way, the quality of the data is defined and the results are of known value. These checks and tests also provide timely and specific warning of any problems which might develop within the sampling and analysis operations. A description of these quality control/quality assurance procedures is summarized in the following Table III:

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TABLE III--SUMMARY OF LABORATORY DATA QUALITY OBJECTIVES

Unit Operation	QC Check	Frequency	Conformance Expectation
Sample receiving	Review of receiving report	Each sample	95% complete
Sample custody	Review of chain-of-custody record	Each sample	95% complete
Sample preparation	Supplies and reagents	On receipt	Meet specs. or reject
	Grid opening size	20 openings/20 grids/lot of 1000 or 1 opening/sample	100%
	Special clean area monitoring	After cleaning or service	Meet specs or reclean
	Laboratory blank	1 per prep series or 10%	Meet specs. or reanalyze series
	Plasma etch blank	1 per 20 samples	75%
	Multiple preps (3 per sample)	Each sample	One with cover of 15 complete grid sqs.
Sample analysis	System check	Each day	Each day
	Alignment check	Each day	Each day
	Magnification calibration with low and high standards	Each month or after service	95%
	ED calibration by gold standard	Weekly	95%
	EDS calibration by copper line	Daily	95%
Performance check	Laboratory blank (measure of cleanliness)	Prep 1 per series or 10% read 1 per 25 samples	Meet specs or reanalyze series
	Replicate counting (measure of precision)	1 per 100 samples	1.5 x Poisson Std. Dev.
	Duplicate analysis (measure of reproducibility)	1 per 100 samples	2 x Poisson Std. Dev.
	Known samples of typical materials (working standards)	Training and for comparison with unknowns	100%
	Analysis of NBS SRM 1876 and/or RM 8410 (measure of accuracy and comparability)	1 per analyst per year	1.5 x Poisson Std. Dev.
	Data entry review (data validation and measure of completeness)	Each sample	95%
	Record and verify ID electron diffraction pattern of structure	1 per 5 samples	80% accuracy
Calculations and data reduction	Hand calculation of automated data reduction procedure or independent recalculation of hand-calculated data	1 per 100 samples	85%

BILLING CODE 6560-50-C

1. When the samples arrive at the laboratory, check the samples and documentation for completeness and requirements before initiating the analysis.

2. Check all laboratory reagents and supplies for acceptable asbestos background levels.

3. Conduct all sample preparation in a clean room environment monitored by laboratory blanks and special testing after cleaning or servicing the room.

4. Prepare multiple grids of each sample.

5. Provide laboratory blanks with each sample batch. Maintain a cumulative average of these results. If this average is greater than 53 f/mm ^{2} per 10 200-mesh grid openings, check the system for possible sources of contamination.

6. Check for recovery of asbestos from cellulose ester filters submitted to plasma asher.

7. Check for asbestos carryover in the plasma asher by including a blank alongside the positive control sample.

8. Perform a systems check on the transmission electron microscope daily.

9. Make periodic performance checks of magnification, electron diffraction and energy dispersive X-ray systems as set forth in Table III of Unit III.K.

10. Ensure qualified operator performance by evaluation of replicate counting, duplicate analysis, and standard sample comparisons as set forth in Table III of Unit III.K.

11. Validate all data entries.

12. Recalculate a percentage of all computations and automatic data reduction steps as specified in Table III.

13. Record an electron diffraction pattern of one asbestos structure from every five samples that contain asbestos. Verify the identification of the pattern by measurement or comparison of the pattern with patterns collected from standards under the same conditions.

The outline of quality control procedures presented above is viewed as the minimum required to assure that quality data is produced for clearance testing of an asbestos abated area. Additional information may be gained by other control tests. Specifics on those control procedures and options available for environmental testing can be obtained by consulting References 6, 7, and 11 of Unit III.L.

L. References

For additional background information on this method the following references should be consulted.

1. "Guidelines for Controlling Asbestos-Containing Materials in Buildings," EPA 560/5–85–024, June 1985.

2. "Measuring Airborne Asbestos Following an Abatement Action," USEPA/ Office of Toxic Substances, EPA 600/4–85–049, 1985.

3. Small, John and E. Steel. Asbestos Standards: Materials and Analytical Methods. N.B.S. Special Publication 619, 1982.

4. Campbell, W.J., R.L. Blake, L.L. Brown, E.E. Cather, and J.J. Sjoberg. Selected Silicate Minerals and Their Asbestiform Varieties. Information Circular 8751, U.S. Bureau of Mines, 1977.

5. Quality Assurance Handbook for Air Pollution Measurement System. Ambient Air Methods, EPA 600/4–77–027a, USEPA, Office of Research and Development, 1977.

6. Method 2A: Direct Measurement of Gas Volume Through Pipes and Small Ducts. 40 CFR Part 60 Appendix A.

7. Burdette, G.J. Health & Safety Exec., Research & Lab. Services Div., London, "Proposed Analytical Method for Determination of Asbestos in Air."

8. Chatfield, E.J., Chatfield Tech. Cons., Ltd., Clark, T., PEI Assoc. "Standard Operating Procedure for Determination of Airborne Asbestos Fibers by Transmission Electron Microscopy Using Polycarbonate Membrane Filters." WERL SOP 87–1, March 5, 1987.

9. NIOSH. Method 7402 for Asbestos Fibers, December 11, 1986 Draft.

10. Yamate, G., S.C. Agarwall, R.D. Gibbons, IIT Research Institute, "Methodology for the Measurement of Airborne Asbestos by Electron Microscopy." Draft report, USEPA Contract 68–02–3266, July 1984.

11. Guidance to the Preparation of Quality Assurance Project Plans. USEPA, Office of Toxic Substances, 1984.

IV. Mandatory Interpretation of Transmission Electron Microscopy Results to Determine Completion of Response Actions

A. Introduction

A response action is determined to be completed by TEM when the abatement area has been cleaned and the airborne asbestos concentration inside the abatement area is no higher than concentrations at locations outside the abatement area. "Outside" means outside the abatement area, but not necessarily outside the building. EPA reasons that an asbestos removal contractor cannot be expected to clean an abatement area to an airborne asbestos concentration that is lower than the concentration of air entering the abatement area from outdoors or from other parts of the building. After the abatement area has passed a thorough visual inspection, and before the outer containment barrier is removed, a minimum of five air samples inside the abatement area and a minimum of five air samples outside the abatement area must be collected. Hence, the response action is determined to be completed when the average airborne asbestos concentration measured inside the abatement area is not statistically different from the average airborne asbestos concentration measured outside the abatement area.

The inside and outside concentrations are compared by the Z-test, a statistical test that takes into account the variability in the measurement process. A minimum of five samples inside the abatement area and five samples outside the abatement area are required to control the false negative error rate, i.e., the probability of declaring the removal complete when, in fact, the air concentration inside the abatement area is significantly higher than outside the abatement area. Additional quality control is provided by requiring three blanks (filters through which no air has been drawn) to be analyzed to check for unusually high filter contamination that would distort the test results.

When volumes greater than or equal to 1.199 L for a 25 mm filter and 2.799 L for a 37 mm filter have been collected and the average number of asbestos structures on samples inside the abatement area is no greater than 70 s/mm ^{2} of filter, the response action may be considered complete without comparing the inside samples to the outside samples. EPA is permitting this initial screening test to save analysis costs in situations where the airborne asbestos concentration is sufficiently low so that it cannot be distinguished from the filter contamination/ background level (fibers deposited on the filter that are unrelated to the air being sampled). The screening test cannot be used when volumes of less than 1.199 L for 25 mm filter or 2.799 L for a 37 mm filter are collected because the ability to distinguish levels significantly different from filter background is reduced at low volumes.

The initial screening test is expressed in structures per square millimeter of filter because filter background levels come from sources other than the air being sampled and cannot be meaningfully expressed as a concentration per cubic centimeter of air. The value of 70 s/mm^2 is based on the experience of the panel of microscopists who consider one structure in 10 grid openings (each grid opening with an area of 0.0057 mm^2) to

be comparable with contamination/background levels of blank filters. The decision is based, in part, on Poisson statistics which indicate that four structures must be counted on a filter before the fiber count is statistically distinguishable from the count for one structure. As more information on the performance of the method is collected, this criterion may be modified. Since different combinations of the number and size of grid openings are permitted under the TEM protocol, the criterion is expressed in structures per square millimeter of filter to be consistent across all combinations. Four structures per 10 grid openings corresponds to approximately 70 s/mm².

B. Sample Collection and Analysis

1. A minimum of 13 samples is required: five samples collected inside the abatement area, five samples collected outside the abatement area, two field blanks, and one sealed blank.

2. Sampling and TEM analysis must be done according to either the mandatory or nonmandatory protocols in Appendix A. At least 0.057 mm² of filter must be examined on blank filters.

C. Interpretation of Results

1. The response action shall be considered complete if either:

a. Each sample collected inside the abatement area consists of at least 1.199 L of air for a 25 mm filter, or 2.799 L of air for a 37 mm filter, and the arithmetic mean of their asbestos structure concentrations per square millimeter of filter is less than or equal to 70 s/mm²; or

b. The three blank samples have an arithmetic mean of the asbestos structure concentration on the blank filters that is less than or equal to 70 s/mm² and the average airborne asbestos concentration measured inside the abatement area is not statistically higher than the average airborne asbestos concentration measured outside the abatement area as determined by the Z-test. The Z-test is carried out by calculating

$$Z = \frac{\bar{Y}_I - \bar{Y}_0}{0.8\left(1/n_I + 1/n_0\right)^{1/2}}$$

where $\bar{Y}_I$ is the average of the natural logarithms of the inside samples and $\bar{Y}_0$ is the average of the natural logarithms of the outside samples, n_I is the number of inside samples and n_0 is the number of outside samples. The response action is considered complete if Z is less than or equal to 1.65.

(Note.—When no fibers are counted, the calculated detection limit for that analysis is inserted for the concentration.)

2. If the abatement site does not satisfy either (1) or (2) above, the site must be recleaned and a new set of samples collected.

D. Sequence for Analyzing Samples

It is possible to determine completion of the response action without analyzing all samples. Also, at any point in the process, a decision may be made to terminate the analysis of existing samples, reclean the abatement site, and collect a new set of samples. The following sequence is outlined to minimize the number of analyses needed to reach a decision.

1. Analyze the inside samples.

2. If at least 1.199 L of air for a 25 mm filter or 2.799 L of air for a 37 mm filter is collected for each inside sample and the arithmetic mean concentration of structures per square millimeter of filter is less than or equal to 70 s/mm², the response action is complete and no further analysis is needed.

3. If less than 1.199 L of air for a 25 mm filter or 2.799 L of air for a 37 mm filter is collected for any of the inside samples, or the arithmetic mean concentration of structures per square millimeter of filter is greater than 70 s/mm², analyze the three blanks.

4. If the arithmetic mean concentration of structures per square millimeter on the blank filters is greater than 70 s/mm², terminate the analysis, identify and correct the source of blank contamination, and collect a new set of samples.

5. If the arithmetic mean concentration of structures per square millimeter on the blank filters is less than or equal to 70 s/mm², analyze the outside samples and perform the Z-test.

6. If the Z-statistic is less than or equal to 1.65, the response action is complete. If the Z-statistic is greater than 1.65, reclean the abatement site and collect a new set of samples.

Appendix B to Subpart E—Work Practices and Engineering Controls for Small-Scale, Short-Duration Operations Maintenance and Repair (O&M) Activities Involving ACM

This appendix is not mandatory, in that LEAs may choose to comply with all the requirements of 40 CFR 763.121. Section 763.91(b) extends the protection provided by EPA in its 40 CFR 763.121 for worker protection during asbestos abatement projects to employees of local education agencies who perform small-scale, short-duration operations, maintenance and repair (O&M) activities involving asbestos-containing materials and are not covered by the OSHA asbestos construction standard at 29 CFR 1926.58 or an asbestos worker protection standard adopted by a State as part of a State plan approved by OSHA under section 18 of the Occupational Safety and Health Act. Employers wishing to be exempt from the requirements of § 763.121 (e)(6) and (f)(2)(i) may instead comply with the provisions of this appendix when performing small-scale, short-duration O&M activities.

Definition of Small-Scale, Short-Duration Activities

For the purposes of this appendix, small-scale, short-duration maintenance activities are tasks such as, but not limited to:

1. Removal of asbestos-containing insulation on pipes.

2. Removal of small quantities of asbestos-containing insulation on beams or above ceilings.

3. Replacement of an asbestos-containing gasket on a valve.

4. Installation or removal of a small section of drywall.

5. Installation of electrical conduits through or proximate to asbestos-containing materials.

Small-scale, short-duration maintenance activities can be further defined, for the purposes of this subpart, by the following considerations:

1. Removal of small quantities of asbestos-containing materials (ACM) only if required in the performance of another maintenance activity not intended as asbestos abatement.

2. Removal of asbestos-containing thermal system insulation not to exceed amounts greater than those which can be contained in a single glove bag.

3. Minor repairs to damaged thermal system insulation which do not require removal.

4. Repairs to a piece of asbestos-containing wallboard.

5. Repairs, involving encapsulation, enclosure or removal, to small amounts of friable asbestos-containing material only if required in the performance of emergency or routine maintenance activity and not intended solely as asbestos abatement. Such work may not exceed amounts greater than those which can be contained in a single prefabricated minienclosure. Such an enclosure shall conform spatially and geometrically to the localized work area, in order to perform its intended containment function.

Appendix 6 - Examples of Forms Used

ASBESTOS AIR SAMPLING RECORD

DATE:

BLDG. & PROJECT NAME:

	SAMPLE I.D. NO.	TYPE *	ACTIVITY**	DESCRIPTION OF SAMPLING LOCATION & COMMENTS
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				

ACCOUNTABILITY RECORD PAGE____ OF____

REQUESTED COMPLETION DATE: _____

JOB NO.:_____ BATCH NO.:_____

CLIENT NAME:_____

SAMPLER'S NAME: _____

SIGNATURE:_____

DATE:_____ TIME COMPLETED:_____

DELIVERED TO LAB BY: _____

LAB NAME:_____

RECEIVED BY: _____

DATE:_____ TIME:_____ INITIALS:_____

ANALYST:_____ SCOPE #:_____

DATE COMPLETED:_____ TIME:_____

ANALYSIS METHOD:_____

APPROVAL SIGNATURE (PROJECT MANAGER): _____

SAMPLING AND ANALYTICAL DATA

EFFECTIVE FILTER AREA =_____ MM ^{2}; FIELD AREA = _____ MM ^{2}

	PUMP I.D. NO.	FLOW METER I.D. NO.	AIR FLOWRATE (LPM) START	AIR FLOWRATE (LPM) STOP	AIR FLOWRATE (LPM) AVERAGE	SAMPLING TIME START	SAMPLING TIME STOP	SAMPLING TIME TOTAL	VOLUME (LITERS)	FIBERS / FIELDS	AVERAGE BLANK COUNT	ANALYST'S CV	LOQ	FIBERS PER CC	UPPER CONFIDENCE LIMIT
1															
2															
3															
4															
5															
6															
7															
8															
9															
10															

SPECIAL COMMENTS:

* SAMPLE TYPE CODES

PCM = PHASE CONTRAST MICROSCOPY
TEM = TRANSMISSION ELECTRON MICROSCOPY
SEM = SCANNING ELECTRON MICROSCOPY
A = PERSONAL EXPOSURE SAMPLE
B = WORK AREA CLEARANCE
C = PREABATEMENT BACKGROUND
D = OUTSIDE WORK AREA
E = INSIDE WORK AREA
F = AIR QUALITY/ENVIRONMENTAL
G = HEPA EXHAUST DISCHARGE
H = DECONTAMINATION FACILITY
I = BLANK SAMPLE

** WORK AREA ACTIVITY CODES

1 = PREABATEMENT BACKGROUND
2 = PREPARATION OF WORK AREA
3 = ASBESTOS REMOVAL WORK
4 = CLEAN UP OF WORK AREA
5 = WASTE REMOVAL
6 = GLOVE BAG REMOVAL WORK
7 = WORK AREA CLEARANCE
8 = MAINTENANCE ACTIVITY
9 = ACBM REPAIR ACTIVITY
10 = ACBM ENCAPSULATION OR ENCLOSURE
11 = CLEANING OR DECONTAMINATION
12 = NOT APPLICABLE

CLIENT BILLING INSTRUCTIONS: _____ SAMPLES @ $_____ = $

CAMERA CONSTANT DETERMINATION

Operator:_____ Negative #_____

Date:_____

Instrument:_____ Projector Setting:_____

Accelerating Voltage: 80kV

	R1	R2	R3	R4
d1				
d2				
d3				
r				
Å	2.355	2.039	1.442	1.230
r x Å				

Average = _____ mmÅ

# of layer lines eclipsed by Objective Lens Aperture #2 : _____

MAGNIFICATION CALIBRATION

Operator: _____ Date: _____
Instrument: _____
Accelerating Voltage: 80kV Neg. # _____

SCREEN MAGNIFICATION

Count the number of spacings/lines on the grating replica within the diameter of the large circle on the screen at~18000x.

$$\frac{\text{Circle diameter X Actual Replica Spacing}}{\text{Number of Spacings/Lines}}$$

$$\frac{(90\text{mm}) \text{ X } (2160 \text{ lines/mm})}{(\_\_\_\_\_ \text{lines})}$$

PLATE MAGNIFICATION

Take a photograph of the grating replica at~18000x. Measure the distance between several lines and count the number of lines within the measured area.

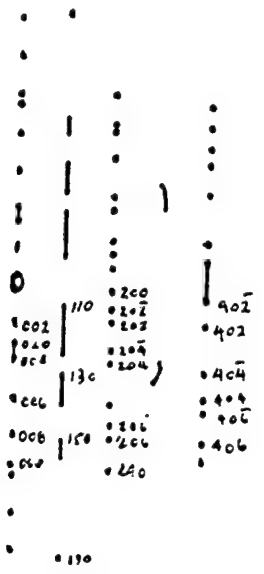
$$\frac{\text{Distance Measured X Actual Replica Spacing}}{\text{Number of Lines Counted}}$$

$$\frac{(\_\_\_\_\_ \text{mm}) \text{ X } (2160 \text{ lines/mm})}{(\_\_\_\_\_ \text{lines})}$$

RESULTS:

Screen Mag = _____x
Scribed circle on screen (Hi Mag): Large: 90000/screen mag = _____um
Small: 9000/screen mag = _____um
Unit on scale bar (Hi Mag): 900/screen mag = _____um,
Entire length of scale ba. (Hi Mag): 18000/screen mag = _____um
Plate Mag = _____x

CHRYSOTILE INDEXING



Camera Constant = _____ mm Å

Measure the following diffraction spots and determine d:

$$d(\text{Å}) = \frac{CC\ (\text{mm Å})}{\text{distance (mm)}}$$

Miller Index	Distance (mm)	d(Å)	ASTM
002	_____	_____	7.31
004	_____	_____	3.65
020	_____	_____	4.50
110	_____	_____	4.55
202	_____	_____	2.45
Row Spacing	_____	_____	5.3

β = _____
(Center to 200)

β = 87°

Beam Dose Calibration

DATE	ANALYST	INSTRUMENT	VOLTAGE	SECONDS	RESULT

GRAVIMETRIC ASHER CALIBRATION

Clean a glass slide and weigh on the analytical balance in the Chemistry Lab. Cut a section of filter using the cork borer, and collapse in an acetone vapor chamber for 4 minutes. (Do not use the page re-inforcer to attach the filter section.) Weigh again. Subtract the weight of the empty slide to obtain the weight of the collapsed filter. Ash the sample for the specified time, and weigh again. Subtract the weight of the empty slide to obtain the weight of the collapsed, etched filter. Subtract the weight of the unetched filter from the etched filter. This difference should reflect a 10 % weight loss during the etching process. If not, the time of etching should be adjusted accordingly, and the above process should be repeated until the time used for etching produces a 10 % weight loss.

DATE: _____
INITIALS: _____
TIME ASHED: _____ minutes

1) Weight of glass slide:_____g
2) Weight of slide plus collapsed, unetched filter section:_____g
3) Weight of slide plus collapsed, etched filter section:_____g

4) Weight of collapsed, unetched filter section (2-1):_____g
5) Weight of collapsed, etched filter section (3-1):_____g
6) Weight difference (4-5):_____g
7) % difference (6/4 x 100):_____%

H&GCL ASBESTOS BULK SAMPLE DATA FORM

BLDG. NAME & ADDRESS:

	SAMPLE I.D. NO.	TYPE*	FLOOR NO.	SAMPLE DESCRIPTION AND LOCATION	FUNCTION CODE	TYPE CODE
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						

ACCOUNTABILITY RECORD

SHEET_____OF_____

REQUESTED COMPLETION DATE:_____

JOB NO.:_____ BATCH NO.:_____

CLIENT NAME:_____

SAMPLER'S NAME: _____

SIGNATURE:_____

DATE:_____ TIME COMPLETED_____

DELIVERED TO LAB BY: _____

LAB NAME: _____

ADDRESS:_____

RECEIVED BY:_____

DATE:_____ TIME:_____ SIGNATURE:_____

ANALYZED BY:_____ DATE:_____

LAB QC APPROVAL: _____

PROJECT MANAGER'S APPROVAL: _____

ANALYTICAL LABORATORY RESULTS

	LAB ID. NO	ASBESTOS CONCENTRATIONS**						CONCENTRATIONS OF OTHER COMPONENTS							SAMPLE COLOR	PC CONC.	NY-PC CONC.	ANALYSIS COMMENTS
		CH	AM	CR	AC	AN	TR	FG	MW	CL	SY	HA	NF	OT				
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		
13																		

ADDITIONAL COMMENTS & NOTES:

*SAMPLE TYPE CODES

B = BULK MATERIAL
D = DEBRIS SAMPLE
SD/T = SURFACE DUST - TAPE SAMPLE
SD/V = SURFACE DUST - VACUUM SAMPLE
SD/G = SURFACE DUST - GRAB SAMPLE

ASBESTOS TYPE CODES

CH = CHRYSOTILE
AM = AMOSITE
CR = CROCIDOLITE
AC = ACTINOLITE
AN = ANTHOPHYLLITE
TR = TREMOLITE

**CONCENTRATIONS DETERMINED BY VISUAL ESTIMATION TECHNIQUE UNLESS OTHERWISE NOTED

ABBREVIATIONS FOR OTHER COMPONENTS

FG = FIBERGLASS
MW = MINERAL WOOL
CL = CELLULOSE
SY = SYNTHETIC FIBER
HA = HAIR
NF = NON-FIBEROUS MATERIAL
OT = OTHER
PC = POINT COUNTING
NY-PC = NY STATE POINT COUNTING

CLIENT BILLING INSTRUCTIONS

NO. SAMPLES	RATE	SUBTOTAL

OUTSIDE LAB AUTHORIZATION

Batch # | Analyst:

Date: | Analyst QC:

Analysis Data Sheet

H+GCL

A Lab ID				
B Macroscopic Exam				
1. Color				
2. Homogenious Y/N				
3. Texture				
4. Fnable Y/N				
5. Fibrous Phases Observed				
6. Number of layers				
7. Tentative ID				
8. Sample Prep: 1-Pulverized 2- Thermal ashing 3- Acid dissolution 4- Other 5- None				
C Microscopic Exam				
9. Type of asbestos	CH | AM | CR | ACT | AN | TR	CH | AM | CR | ACT | AN | TR	CH | AM | CR | ACT | AN | TR	CH | AM | CR | ACT | AN | TR
10. Morphology				
11. Extinction || or ⊿				
12. Sign of elongation +/-				
13. Birefringence L / M / H				
14. Pleochroism Y/N				
15. RI / DS 1.55: 1.68 || 1.605; Other ⊥				
D Other Fibers	FG | MW | CL | SYN | HA | OTH	FG | MW | CL | SYN | HA | OTH	FG | MW | CL | SYN | HA | OTH	FG | MW | CL | SYN | HA | OTH
16. Morphology				
17. Sign of elongation + / - / D				
18. Isotropic Y/N				
19. Cleavage				
20. Relief L / M / H				
21. Birefringence L / M / H				
22. Scaly growth pattern Y/N				
23. Non fibrous				
24. Comments				

Texture:
P = Powdery
F = Fibrous
GN = Granular
OTH = Describe

Sample Prep:
OTH = Describe

Morphology:
W = Wavy
S = Straight
AC = Acicular
PR = Prismatic
FL = Flat
CY = Cylindrical
CU = Curly
NL = Needle Like

Sign of Elongation:
D = Double

Colors:
BL = Blue
YL = Yellow
GR = Green
RD = Red
BR = Brown
MG = Magenta
BK = Black
TN = Tan
GY = Gray
LT = Light
DK = Dark

Batch # | Analyst:

Date: | Analyst QC:

QC Analysis Data Sheet

H+GCL

A Lab ID				
B Macroscopic Exam				
1. Color				
2. Homogenious Y/N				
3. Texture				
4. Friable Y/N				
5. Fibrous Phases Observed				
6. Number of layers				
7. Tentative ID				
8. Sample Prep: 1-Pulverized 2- Thermal ashing 3- Acid dissolution 4- Other 5- None				
C Microscopic Exam	CH AM CR ACT AN TR	CH AM CR ACT AN TR	CH AM CR ACT AN TR	CH AM CR ACT AN TR
9. Type of asbestos				
10. Morphology				
11. Extinction || or ⊥				
12. Sign of elongation +/-				
13. Birefringence L / M / H				
14. Pleochroism Y/N				
15. RI / DS 1.55: 1.68 ||				
1.605: Other ⊥				
D Other Fibers	FG MW CL SYN HA OTH	FG MW CL SYN HA OTH	FG MW CL SYN HA OTH	FG MW CL SYN HA OTH
16. Morphology				
17. Sign of elongation + / - / D				
18. Isotropic Y/N				
19. Cleavage				
20. Relief L / M / H				
21. Birefringence L / M / H				
22. Scaly growth pattern Y/N				
23. Non fibrous				
24. Comments				

Texture:
P = Powdery
F = Fibrous
GN = Granular
OTH = Describe

Sample Prep:
OTH = Describe

Morphology:
W = Wavy
S = Straight
AC = Acicular
PR = Prismatic
FL = Flat
CY = Cylindrical
CU = Curly
NL = Needle Like

Sign of Elongation:
D = Double

Colors:
BL = Blue
YL = Yellow
GR = Green
RD = Red
BR = Brown
MG = Magenta
BK = Black
TN = Tan
GY = Gray
LT = Light
DK = Dark
WH = White

Lab ID ______ Method: PC; SPC; SO; % Asb ______

1. 2. 3. 4.

5. 6. 7. 8.

PC = Point countin
SPC = Stratified Pc
SO = Scanning o
Empty spots = NF
x = Asbestos points

Formula:
# of asbestos points
total # of nonempty p

use the same formu
other fibers or partic

Lab ID ______ Method: PC; SPC; SO; % Asb ______

1. 2. 3. 4.

5. 6. 7. 8.

Lab ID ______ Method: PC; SPC; SO; % Asb ______

1. 2. 3. 4.

5. 6. 7. 8.

Lab ID ______ Method: PC; SPC; SO; % Asb ______

1. 2. 3. 4.

5. 6. 7. 8.

Appendix 7 - Other Laboratory Standard Operating Procedures

Standard Operating Procedure For NOB Samples

Introduction

Detection of asbestos in non-friable, organically-bound (NOB) samples is often difficult due to small fiber sizes and interference with matrix material. The following procedure reduces these interferences to allow PLM or TEM identification and quantification of any asbestos present. The TEM method described herein is the only procedure that should be used to report a true negative of NOB samples.

Equipment and Supplies

Analytical balance
muffle furnace
stereoscope
PLM
TEM w/EDAX
fume hood
mini-blender
hot plate
filtration set up
ultrasonic bath
micropipette
acetone
conc. HCl
dist. water
gloves
goggles
scalpel
tweezers
mortar and pestle
0.4 um polycarb. filters
petri dishes
crucibles w/lids
disposable beakers
glass slides
coverslips
dispersion oils
formvar grids
ethanol

Sample Preparation Procedure

Using a separate analysis sheet for each sample as a guide, prepare the samples as follows:

1. Carefully scrape away or dissolve any mastic or otherwise extraneous material attached to the sample.
2. Shave approximately 100-500 mg of the sample into a preweighed crucible and weigh. Do not use the hood in the TEM area for this process.
3. Place crucible with lid in muffle furnace at 480°C for about 12 hours. At least 12 crucibles will fit into our muffle furnace. Be sure that the hood is vented to the outside to avoid breathing potentially harmful emissions from samples as they volatilize.
4. Allow samples to cool and weigh.
5. Grind residue in the crucible with a pestle and 0.5ml distilled water. Rinse pestle briefly into crucible to remove any visible debris.
6. Wearing protective gloves and goggles, add 2-5ml concentrated HCl and allow to stand for about 15 minutes.
7. Set up filtration apparatus with a preweighed PC filter and 50ml distilled water. Pour contents of crucible into funnel and rinse crucible so that all debris is flushed into the funnel. Turn on the pump and allow to draw through completely. Perform filtration in a hood if possible, avoiding any vapors. Do not rinse the walls of the funnel after filtration in order to avoid

disturbance of the particle distribution on the filter.
NOTE: Between successive filtrations, all associated glassware and tools should be cleaned in soapy water and rinsed with distilled water.
8. Place filter in a pre-weighed petri dish and allow to dry compltetly.
9. Weigh the filter with the residue.
10. Cut about 1/4 of the filter and transfer to a disposable plastic beaker.
11. Add about 5ml ethanol to the beaker and sonicate for about one minute.
12. While the sample is sonicating, set up 2 formvar grids to receive a drop of sample.
13. Draw about 3ul from the beaker while it is still sonicating using a micropipette and drop mount the liquid onto the grid.
14. When dry, place in a labelled grid box.

Sample Analysis

1. Scan each grid at low magnification (~3000X) to ensure that the particle distribution is relatively even across the grid and that there is about 10-50% particle loading.
2. If the grid is acceptable, increase magnification to ~20000X to identify the particles present. If any of the asbestos minerals are present, identify each species using morphology, crystal structure and chemistry.
3. Estimate the percentage of each asbestos type present in the residue and note any other non-asbestos particles present by their chemistry.
4. If the sample is negative by negative by TEM, and has not previously been subjected to PLM analysis, the residue on the filter must be analyzed by PLM. Two coverslip preparations should be made using about 1/4 of the residue.
5. If any traces of asbestos are found, 2 additional preparations must be made and a mean percentage estimate of the four samples should be used for the calculations.

Calculations

All calculations used are found on the sample analysis sheet attached to this SOP.

Bulk Sample Analysis Sheet

Analyst:_____ Analysis Date:_____ Sample ID:_____

STEREOBINOCULAR MICROSCOPY:

Color:_____ Texture:_____ Homogeneity:_____ Homogenization:_____

Probable Fibers:_____ Remarks:_____

MATRIX REDUCTION:

ASHING: mg: A:(Crucible)_____ B:(Crucible + Subsample)_____ C:(Crucible + Ashed Subsample)_____

ACID DIGESTION: mg: D:(Filter/Petri)_____ E:(Filter/Petri + Residue)_____

CALCULATIONS	mg		Percent	
	Calculation	Result	Calculation	Result
Untreated Sample	(F = B - A)		(G = 100%)	100%
Organic Component	(H = (F - (C - A))		(I = (H X G) / F)	
Acid-Insoluble Inorganic Component	(J = E - D)		(K = (J X G) / F)	
Acid-Soluble Inorganic Component	(L = (F - (J + H)))		(M = (L X G) / F)	

Remarks:_____

TRANSMISSION ELECTRON MICROSCOPE:

IDENTIFICATION: Storage Grid Box/Slot: Grid 1:_____ / Grid 2:_____ /

Grid	Morphology	Electron Diffraction Observations	X-Ray Observation	Remarks	Identity

QUANTITATION:

Estimated Percent Asbestos on: Grid 1:_____ Grid 2:_____ Mean (N):_____

Percent Asbestos in Subsample: (N X K)/G:_____

use only if sample is req by TEM & has not already been an. by PLM

POLARIZED-LIGHT MICROSCOPE:

IDENTIFICATION:_____

QUANTITATION: (N):_____ Remarks:_____

Percent Asb in Subsample: (N x K) G:_____

FINAL RESULTS:

COMPONENT:	ASBESTOS TYPE _____	ASBESTOS TYPE _____	ORGANIC	ACID-SOLUBLE INORGANIC	INSOLUBLE NON-ASBESTOS INORGANIC
PERCENT:					

Verified:_____ Date:_____ Remarks:

Standard Operating Procedure For TEM Quick Prep Samples

Introduction

Detection of asbestos in non-friable, organically-bound samples is often difficult due to small fiber sizes and interference with matrix material. The following procedure allows a qualitative determination of the presence of asbestos in samples that may have been analyzed by PLM and found to be negative. The TEM method described herein is to be used as a "yes/no" method. The full NOB method (or Chatfield Method) should be used to report a true negative of NOB samples.

Equipment and Supplies

scalpel
TEM w/EDAX
fume hood
hot plate
ultrasonic bath
tweezers
micropipette
formvar grids
disposable test tubes
acetone

Sample Preparation Procedure

All preparation of samples is to be performed in an area separate from the TEM Air prep area until the samples are fully immersed in acetone and ready for sonication.

1. Carefully scrape away or dissolve any mastic or otherwise extraneous material attached to the sample.
2. Shave approximately 100mg of the sample into a clean, disposable test tube. Do not use the hood in the TEM area for this process.
3. Add about 5ml acetone to the test tube. Cover lightly and sonicate for about one minute.
4. While the sample is sonicating, set up a formvar grid to receive a drop of sample.
5. Draw about 3ul from the test tube after the sample appears to have dissolved significantly using a micropipette and drop mount the liquid onto the grid.
6. When dry, place in a labelled grid box.

Sample Analysis and Reporting

1. Scan each grid at low magnification (~3000X) to ensure that the particle distribution is relatively even across the grid and that there is about 10-50% particle loading.
2. If the grid is acceptable, increase magnification to ~20000X to identify the particles present. If any of the asbestos minerals are present, identify each species using morphology, crystal structure and chemistry and note the type(s) of asbestos present.
3. Report samples as being positive for asbestos or no asbestos detected. This method is not to be used quantitatively.

Standard Operating Procedure For Dust Samples By TEM

Introduction

This test method is to be used to test settled dust samples for asbestos. The sample may be collected from furniture, carpets, tops of ceiling tiles, or any other surface suspected to be contaminated. This technique augments other methods in the evaluation of potential health hazards associated with the use, maintenance or cleaning of the surfaces tested. The result of the method is quantitative, but the use of the results is usually in comparison to known clean areas. This method also does not determine the absolute "cleanliness" of a surface, or the release potential of the asbestos.

Equipment and Supplies

TEM w/EDAX
carbon evaporator
clean bench (HEPA)
ultrasonic bath
sample containers
filtration set-up
tweezers
glass petri dishes
particle-free water
scalpel
glass slides
analyslides
0.22um MCE filters
5.0um MCE filters
micropipette
hot plate
TEM grids
acetone

Sample Preparation Procedure

All sample preparation must be performed in a clean area and within the guidelines of HGCL Analytical Services Laboratory's QA/QC Procedures Manual.

1. Clean the exterior portion of the cassette by wet wiping to avoid contaminating the clean area.
2. Remove the cap from the cassette and add approximately 10ml of water. If PVC tubing was included with the sample, rinse it with water into the cassette.
3. Replace the cap and shake for 15 to 30 seconds.
4. Pour the contents of the cassette into the sample container and rinse the cassette thoroughly into the container so that no visible residue remains in the cassette. Repeat for a total of three washings. Rinse the nozzle 2 or 3 times into the container, and then bring the total volume to 100ml.
5. Set up the filtration apparatus with a 0.22um filter and a 5um backing filter. Before applying vacuum, add 20ml water to funnel.
6. Shake the dust suspension for 30 seconds and ultrasonicate for 10 minutes. Allow the suspension to sit for 2 minutes to allow large particles to settle out.
7. Estimate the amount of sample required to leave a light stain on the filter. Depending upon the concentration of dust, this could be anywhere from 1 to 50 ml. With a precleaned pipette, withdraw the aliquot and introduce it to the funnel.
8. Draw the liquid through the filter. Several aliquots may need to be drawn in order to produce optimal sample loading.

9. Disassemble the apparatus and remove the filter with a tweezers. Place the filter in a plastic analyslide and allow to dry. This process will go more quickly if a hot plate is used.
10. When dry, the samples are prepared as a typical air sample - a filter wedge is cut, collapsed, lightly ashed, carbon coated and prepped on a Jaffe Wick. This process is described in detail in the QA/QC Manual.

Sample Analysis Procedure

The sample analysis procedure is very similar to that of an air sample (including grid acceptability criteria, structure classification, identification of asbestos fibers, etc.). The main differences are as follows:
1. Each fiber must be measured (length x width).
2. Large clusters should be classified into one of three categories based on the number of fibers present: <10, 10-100, >100
3. The stopping rule is the completion of the grid square opening that achieves an analytical sensitivity of 162 asbestos structures per square centimeter. If 162 str/sq. cm cannot be reached in 21 grid square openings, stop on the 21st opening or the grid opening which contains the 100th structure, whichever comes first. A minimum of 10 grid openings should be counted.

Sample Reporting Information

The following information must be reported for each dust sample analyzed:
1. Concentration of asbestos (structures per square centimeter).
2. Type(s) of asbestos present.
3. Number of asbestos structures counted.
4. Area of the filter analyzed.
5. Area of the surface analyzed.
6. Structure size data.
7. Name of analyst.

Standard Operating Procedure For Point Counting

Introduction

Quantification of asbestos in bulk samples can be subjective, especially in samples containing low percentages. The following procedure reduces subjectivity in the quantification of asbestos in PLM samples and should be used (with the client's consent) for samples in the 1%-10% range.

Equipment and Supplies

stereoscope
PLM
HEPA hood
hot plate
counting device
dispersion oils
scalpel
tweezers
mortar and pestle
glass slides and coverslips
acetone
point counting reticle

Sample Preparation Procedure

Perform all sample manipulations inside a HEPA filtered hood. Using a point count analysis sheet for each sample as a guide, prepare the samples as follows:

1. Be sure the sample is dry before beginning the analysis. If not, dry the sample on the hot plate.
2. Homogenize the sample if necessary using a mortar and pestle.
3. At least 8 subsamples must be mounted on labelled glass slides in a random method (do not bias the sample by choosing mostly fibrous or nonfibrous material). Usually, 2 subsamples can prepared on the same slide under separate coverslips.

Sample Analysis

Perform analysis of samples at 100X, and unless otherwise warranted, under crossed polars and with the first-order red compensator plate inserted. A point is the intersection of two mutually perpendicular lines in the eyepiece reticle. A nonempty is when a particle coincides with a point. Each nonempty point must be categorized as a specific asbestos variety, a non-asbestos fiber or non-fibrous. An ideal preparation should contain about 50% nonempty points.

1. Moving between fields of view in a random manner, count 50 nonempty points on each of the 8 preps.
2. Keep a tally of each type of point counted and total number of points counted. If a point falls on the intersection of two types of material, score a point for each type of material.
3. Continue counting until 400 nonempty points have been counted
4. The percent asbestos is calculated as follows:

% asbestos = # asbestos counts/# nonempty points X 100

Standard Operating Procedure For Water Samples By TEM

Introduction

This test method is to be used to test potable water samples for asbestos. The sample is usually collected by the client and comes in a one liter glass container. Results of the analysis are reported in mfl (million fibers per liter). EPA guidelines specify 7mfl as the action level for asbestos in drinking water. Sludge and other non-potable water samples may be prepared and analyzed using this method by taking a known aliquot from the original sample and diluting to one liter.

Equipment and Supplies

TEM w/EDAX
carbon evaporator
clean bench (HEPA)
ultrasonic bath
sample containers
filtration set-up
tweezers
glass petri dishes
particle-free water
scalpel
glass slides
analyslides
0.22um MCE filters
5.0um MCE filters
micropipette w/tips
hot plate
TEM grids
acetone

Sample Preparation Procedure

All sample preparation must be performed in a clean area and within the guidelines of HGCL Analytical Services Laboratory's QA/QC Procedures Manual.

1. Set up the filtration apparatus with a 0.22um filter and a 5um backing filter. Before applying vacuum, add 20ml water to funnel.
2. Shake the suspension for 30 seconds and ultrasonicate for 10 minutes.
3. Estimate the amount of sample required to leave a light stain on the filter. Depending upon the concentration of water, this could be anywhere from 1 to 100 ml. With a micropipette, withdraw the aliquot and introduce it to the funnel.
4. Draw the liquid through the filter. Several aliquots may need to be drawn in order to produce optimal sample loading.
5. Disassemble the apparatus and remove the filter with a tweezers. Place the filter in a plastic analyslide and allow to dry. This process will go more quickly if a hot plate is used.
6. When dry, the samples are prepared as a typical air sample - a filter wedge is cut, collapsed, lightly ashed, carbon coated and prepped on a Jaffe Wick. This process is described in detail in the QA/QC Manual.

Sample Analysis Procedure

The sample analysis procedure is very similar to that of an air sample (including grid acceptability criteria, structure classification, identification of asbestos fibers, etc.). The main differences are as follows:

1. Each fiber must be classified into one of two categories: those greater than and those less than 10 microns in length.
2. Continue analysis until 100 fibers are counted or a sensitivity of at least 0.34 mfl is reached. (Generally, 20 grid square openings are counted.)

Sample Reporting Information

The following information must be reported for each water sample analyzed:

1. Concentration of asbestos in mfl for all asbestos and for fibers greater than 10 microns.
2. Type(s) of asbestos present.
3. Number of asbestos structures counted.
4. Analytical sensitivity.
5. Name of analyst.

Calculations

Dilution Factor:

(Used only in the case of Sludge or Non-potable water samples)
For __x__ ml sample added to __y__ ml distilled water:

$$\text{Dilution Factor} = x/y$$

Sensitivity:

$$\frac{\text{Area of Filter (sq. mm)}}{\#\text{GSO} \times \text{GSO Area (sq. mm)} \times \text{ml drawn} \times \text{Dilution Factor}}$$

Area of Filter is usually 1134.1 sq. mm. To obtain units of mfl, move the decimal point three places to the left.

Concentration:

Multiply the number of fibers counted by the sensitivity to obtain the concentration. Perform this calculation for all fibers and for fibers greater than 10 microns.

Standard Operating Procedures for Lab Adminstration

Introduction

In order to keep the sample backlog and invoicing organized, when samples come through the door, they must be placed in the sample tracking system immediately in order to avoid delays in analysis and invoicing.

Internal Clients (Branch Offices)
Samples from branch offices should be checked to be sure that all pertinent information (especially a project number, batch number and date required) is on the form and then logged into the computer (either PLM, PCM or TEM).

External Clients
All external clients must be assigned a project number if one does not already exist. The information should be entered into the white External Client Book kept on top of the black file cabinets in the reception area. The samples associated with that batch should then be logged into the batch book. Initiate an HGCL sample data form if one does not already exist. Once the samples have been assigned a batch number (and project number if necessary), they should be logged into the computer (either PLM, PCM or TEM) so that they may be analyzed without delay.

Once a project number is initiated, a file should be started to hold copies of correspondence, chain of custody forms, copies of reports and invoices, etc. The file should be labelled with the Client's name and project number and filed alphabetically in the black file cabinet in the reception area. A project record form must also be written and sent to Marianne in Accounting.

Invoicing
All internal clients are invoiced through corporate accounting. External clients, however, must be invoiced through the Laboratory. Once a report for a specific batch is completed and signed, an invoice should be typed up and sent out along with the original report. A copy of the report, HGCL sample data sheet and invoice should be filed in the client folder. The original sample data sheet should be filed in numerical order in the external client section of the main filing area. In addition, a copy of the invoice must be sent to Marianne in Accounting. If the samples were accompanied by a check, send the check along with the invoice to Accounting. Obviously, in this case the client would not be sent an invoice; instead file the original invoice in the client folder.

Appendix 8 - Sample Personnel Record

Name: John Smith

Position/Title: Analyst/Member Technical Staff

Areas of Specialization: Phase Contrast Microscopy
Polarized Light Microscopy

Report to: Mary Jones

Responsibilities:

Primary: Perform analyses of bulk samples using light microscopy with the methods prescribed by NBS and EPA Interim Method for Determination of Asbestos in Bulk Insulation Samples - EPA 600/M4-82-020. Perform analyses of air samples using phase contrast microscopy method 7400.

Secondary: Assist the Laboratory Director and PCM/Bulk Analysis Manager with administrative duties that may include logging samples or results into the computer.

Resume of Qualifications: (Include Certificates of Achievement)
(Enclosed)

Appendix B: Balsam/Dames & Moore Quality Assurance Plan

1.0 Introduction

As with any good program, a measurable quality assurance program is necessary to ensure that the end product provides the Army with lead-based paint (LBP) survey data that is reproducible and accurate. In order to provide this assurance, the following two-part quality assurance program will be incorporated into Balsam's LBP survey protocol. This protocol is provided in the Work Plan.

1.1 Organization

Mr. Randall Ames of Balsam will be the Project Manager and coordinator of the lead based paint quality control program. Mr. Ames will work closely with Ms. Erica Cahill and Mr. Richard Waterman of Arthur D. Little to ensure the lead paint sampling program meets USAEC specifications.

2.0 Equipment Quality Control

The primary instrument used to generate the LBP survey data is an x-ray fluorescence (XRF) spectrum analyzer. The instrument used will be a Niton XL spectrum analyzer. This instrument determines the percentage of lead in paint on painted surfaces. To ensure the instruments accuracy, several calibration tests are routinely conducted. At the start and end of any testing day or shift, the XRF instrument is checked against one or more National Institute of Standards and Testing (NIST) SRM 2579 paint standards. These paint standards contain known levels of lead and are used as a reference for checking the calibration and accuracy of the XRF instrument. In addition to calibration checks at the start and end of each survey shift, at least every hour, or whenever the XRF instrument is turned off and then turned back on, it will again be checked against the NIST paint standards. All calibration readings will be recorded in a bound instrument calibration log that will be kept with the instrument and available for review, if requested. If the XRF instrument readings vary from these standards by more than ±0.20 milligrams per square centimeter (mg/cm^2), the unit will not be used until it is re-calibrated.

As an additional confirmation of the accuracy of the XRF instrument, paint chip samples will be collected on a random basis at a frequency of 3 to 5 samples per week. These samples will be analyzed by atomic absorption spectrophotometry (AA) according to the National Institute for Occupational Safety and Health (NIOSH) Method 7082 by ESA Laboratories, Chelmsford, Massachusetts. Standard chain-of-custody forms provided by ESA Laboratories will be used to track all samples. These AA results will be compared to those obtained with the XRF instrument for the same tested surface. If at any time, results vary by more than ±10 percent, additional paint chip testing will be undertaken to verify results. The XRF instrument will be factory checked and re-calibrated if additional paint chip testing still varies by more than ±10 percent.

3.0 Data Quality Control

Lead-paint survey data will be collected by Balsam in two forms. The XRF instrument has an on-board computer that is capable of storing approximately 500 individual test results. The instrument software allows the storage of data including the type of room (kitchen, bath, hall, etc.), the type of surface is being tested (wood, metal, concrete, etc.), the architectural name of the item tested (door/window casings, door, window, floor, etc.), the condition of the paint, the measured lead level(s), and the location in the room which corresponds to wall identifiers shown on the attached field data form. Data collected in the instrument's on-board computer will also be recorded manually on the field data form to provide a paper backup to the computer-stored information. Paint chip samples will also be included with the sample database information.

On a daily basis, data from the XRF instrument's computer will be downloaded to its companion software on an IBM-compatible computer to be maintained at the project site. This data will then be transferred to the database files, also located in the on-site computer. A report will be generated at least weekly and reviewed against the written data forms to verify the computer data. In the event of a difference in data, the written data forms will be considered correct and the database will be corrected to reflect the written data form information. Downloading of the on-site computer database will be done weekly to diskette, one copy to be kept by Balsam and one copy to be give to ADL. Arthur D. Little will maintain the database in a format compatible with dBase IV.